

**HQ Air Force Center for Environmental Excellence  
Technical Services Quality Assurance Program**

**GUIDANCE FOR CONTRACT DELIVERABLES**

**APPENDIX C:  
QUALITY ASSURANCE PROJECT PLAN (QAPP)**



**Version 3.1**

**August 2001**

## **PREFACE**

This document is the *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP)*, version 3.1. This detailed QAPP, (1) has been prepared for use by contractors who perform environmental services to ensure the data are scientifically valid and defensible, and (2) establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner. This QAPP and a site specific Field Sampling Plan (FSP) shall constitute, by definition, an AFCEE Sampling and Analysis Plan (SAP). All prime contractors and laboratories performing work in support of AFCEE contracts shall perform their services in accordance with the requirements specified in this QAPP. A variance shall be requested for any exception to or deviation from the requirements in this QAPP. Variance requests are submitted as an addendum to the SAP. Variances from the QAPP shall be identified by chapter, subtitle, paragraph, page, and line with supporting justification for the change. The original text in this QAPP is crossed out and a reference to the appropriate variance request by number in the addendum is added to the QAPP. If any additional analytical methods are required in the SAP that are not in this QAPP, the analytical methods must be included in the addendum to the SAP with all the accompanying quality control requirements, i.e., reporting limits, calibration requirements, quality control measures, corrective action, data validation, and reporting requirements, comparable in format to the analytical tables in Sections 6, 7, and 8. Variances must be approved by the AFCEE Team Chief for the project. Only the variances approved by the AFCEE Team Chief shall be included in the final version of the SAP.

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## **LIST OF ACRONYMS AND ABBREVIATIONS**

<b>AA</b>	atomic absorption
<b>AAB</b>	AFCEE Analytical Batch
<b>AFCEE</b>	Air Force Center for Environmental Excellence
<b>AFID</b>	Air Force installation identification
<b>A2LA</b>	American Association for Laboratory Accreditation
<b>ARAR</b>	applicable or relevant and appropriate requirement
<b>ASCII</b>	American Standard Code Information Interchange
<b>ASTM</b>	American Society for Testing and Materials
<b>BFB</b>	bromofluorobenzene
<b>Br<sup>-</sup></b>	bromide
<b>BTEX</b>	benzene, toluene, ethylbenzene, xylene
<b>°C</b>	degrees Celsius
<b>CCC</b>	calibration check compound
<b>CERCLA</b>	Comprehensive Environmental Response, Compensation, and Liability Act
<b>CF</b>	calibration factor
<b>CFR</b>	Code of Federal Regulation
<b>Cl<sup>-</sup></b>	chloride
<b>CL</b>	control limit
<b>CLP</b>	Contract Laboratory Program
<b>COC</b>	chain of custody
<b>2,4-D</b>	2,4-dichlorophenoxy propanoic acid
<b>2,4-DB</b>	2,4-dichlorophenoxy butyric acid
<b>DCA</b>	dichloroethane
<b>DCB</b>	dichlorobenzene
<b>DCBP</b>	decachlorobiphenyl
<b>DCE</b>	dichloroethene
<b>DDD</b>	dichlorodiphenyldichloroethane
<b>DDE</b>	dichlorodiphenyldichloroethene
<b>DDT</b>	dichlorodiphenyltrichloroethane
<b>DEQPPM</b>	Defense Environmental Quality Program Policy Memorandum
<b>DFTPP</b>	decafluorotriphenylphosphine
<b>DNB</b>	dinitrobenzene
<b>DNT</b>	dinitrotoluene
<b>DoD</b>	Department of Defense

<b>DQO</b>	data quality objective
<b>DRO</b>	diesel range organics
<b>EDB</b>	ethylene dibromide
<b>EICP</b>	extracted ion current profile
<b>EPA</b>	Environmental Protection Agency
<b>ERPIMS</b>	Environmental Resources Program Information Management System
<b>F<sup>-</sup></b>	fluoride
<b>FID</b>	flame ionization detector
<b>FLAA</b>	flame atomic absorption
<b>FS</b>	feasibility study
<b>FSP</b>	field sampling plan
<b>g</b>	gram
<b>G</b>	glass
<b>GC</b>	gas chromatography
<b>GC/MS</b>	gas chromatography/mass spectroscopy
<b>GFAA</b>	graphite furnace atomic absorption
<b>GRO</b>	gasoline range organics
<b>GCD</b>	<i>Guidance for Contract Deliverables, Version 1.1, March 1998</i>
<b>HCl</b>	hydrochloric acid
<b>HECD</b>	(Hall) electrolytic conductivity detector
<b>HpCDD</b>	heptachlorodibenzo-p-dioxin
<b>HpCDF</b>	heptachlorodibenzofuran
<b>HxCDD</b>	hexachlorodibenzo-p-dioxin
<b>HxCDF</b>	hexachlorodibenzofuran
<b>HMX</b>	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
<b>HNO<sub>3</sub></b>	nitric acid
<b>HPLC</b>	high-performance liquid chromatography
<b>H<sub>2</sub>SO<sub>4</sub></b>	sulfuric acid
<b>IAW</b>	in accordance with
<b>ICP</b>	inductively coupled plasma
<b>ICPES</b>	inductively coupled plasma emission spectroscopy
<b>ICP-MS</b>	inductively coupled plasma - mass spectroscopy
<b>ICS</b>	interference check standard
<b>ID</b>	identification
<b>IRP</b>	Installation Restoration Program
<b>IS</b>	internal standard

<b>LCL</b>	lower control limit
<b>LCS</b>	laboratory control sample
<b>MCPA</b>	(4-chloro-2-methylphenoxy) acetic acid
<b>MCPP</b>	2-(4-chloro-2-methylphenoxy) propionic acid
<b>MDL</b>	method detection limit
<b>mg/kg</b>	milligrams per kilogram
<b>mg/L</b>	milligrams per liter
<b>mL</b>	milliliter
<b>mm</b>	millimeter
<b>MS</b>	matrix spike
<b>MSD</b>	matrix spike duplicate
<b>N/A</b>	not applicable
<b>Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub></b>	sodium thiosulfate
<b>NCP</b>	National Contingency Plan
<b>ng/L</b>	nanograms per liter
<b>ng/mL</b>	nanograms per milliliter
<b>NIST</b>	National Institute of Standards and Technology
<b>nm</b>	nanometer
<b>NO<sub>2</sub><sup>-</sup></b>	nitrite
<b>NO<sub>3</sub><sup>-</sup></b>	nitrate
<b>NTU</b>	nephelometric turbidity unit
<b>OCDD</b>	octachlorodibenzo-p-dioxin
<b>ORP</b>	oxidation-reduction potential
<b>OVA</b>	organic vapor analyzer
<b>P</b>	polyethylene
<b>PAH</b>	polynuclear aromatic hydrocarbon
<b>PCB</b>	polychlorinated biphenyl
<b>PCDD</b>	polychlorinated dibenzo-p-dioxin
<b>PCDF</b>	polychlorinated dibenzofuran
<b>PE</b>	performance evaluation
<b>PeCDD</b>	pentachlorodibenzo-p-dioxin
<b>PeCDF</b>	pentachlorodibenzofuran
<b>PID</b>	photoionization detector
<b>PO<sub>4</sub><sup>-3</sup></b>	phosphate
<b>ppb</b>	parts per billion
<b>ppm</b>	parts per million

<b>ppmv</b>	parts per million volume
<b>PQL</b>	practical quantitation limit
<b>QA</b>	quality assurance
<b>QAPP</b>	quality assurance project plan
<b>QC</b>	quality control
<b>R</b>	recovery
<b>RCA</b>	recommendations for corrective action
<b>RCRA</b>	Resource Conservation and Recovery Act
<b>RDX</b>	hexahydro-1,3,5-trinitro-1,3,5-triazine
<b>RF</b>	response factor
<b>RI</b>	remedial investigation
<b>RI/FS</b>	remedial investigation/feasibility study
<b>RPD</b>	relative percent difference
<b>RSD</b>	relative standard deviation
<b>S</b>	soil
<b>SAP</b>	sampling and analysis plan
<b>SARA</b>	Superfund Amendments and Reauthorization Act
<b>SO<sub>4</sub><sup>-2</sup></b>	sulfate
<b>SOP</b>	standard operating procedure
<b>SOW</b>	statement of work
<b>SPCC</b>	system performance check compound
<b>SVOC</b>	semivolatile organic compound
<b>2,4,5-T</b>	2,4,5-trichlorophenoxy acetic acid
<b>T</b>	California brass
<b>TCA</b>	trichloroethane
<b>TCDD</b>	tetrachlorodibenzo-p-dioxin
<b>TCDF</b>	tetrachlorodibenzofuran
<b>TCE</b>	trichloroethene
<b>TCLP</b>	toxicity characteristic leaching procedure
<b>TCMX</b>	tetrachlorometaxylene
<b>TIC</b>	tentatively identified compound
<b>TNB</b>	trinitrobenzene
<b>TNT</b>	trinitrotoluene
<b>2,4,5-TP</b>	2,4,5-trichlorophenoxy propanoic acid (silvex)
<b>TPH</b>	total petroleum hydrocarbon
<b>UCL</b>	upper control limit

<b>VOC</b>	volatile organic compound
<b>v/v</b>	volume to volume
<b>W</b>	water

## **SYMBOLS**

<b>mg/kg</b>	micrograms per kilogram
<b>mg/L</b>	micrograms per liter
<b>mg/mL</b>	micrograms per milliliter
<b>mL</b>	microliter
<b>mm</b>	micrometer



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## **TABLE OF CONTENTS**

<b>SECTION</b>	<b>PAGE</b>
1.0 Introduction	1-1
2.0 Project Description	2-1
2.1 The U.S. Air Force Installation Restoration Program	2-1
2.2 Purpose and Scope	2-2
2.3 Project Background	2-2
2.4 Project Scope and Objectives	2-2
3.0 Project Organization and Responsibility	3-1
4.0 Quality Program and Data Quality Objectives	4-1
4.1 Data Categories	4-1
4.2 Precision, Accuracy, Representativeness, Completeness, and Comparability	4-1
4.2.1 Precision	4-2
4.2.2 Accuracy	4-2
4.2.3 Representativeness	4-3
4.2.4 Completeness	4-3
4.2.5 Comparability	4-3
4.3 Method Detection Limits, Reporting Limits, and Instrument Calibration Requirements	4-6
4.3.1 Method Detection Limits	4-6
4.3.2 Reporting Limits	4-7
4.3.3 Instrument Calibration	4-7
4.4 Elements of Quality Control	4-8
4.4.1 Laboratory Control Sample	4-9
4.4.2 Matrix Spike/Matrix Spike Duplicate	4-9
4.4.3 Surrogates	4-10
4.4.4 Internal Standards	4-11
4.4.5 Retention Time Windows	4-11

<b>SECTION</b>	<b>PAGE</b>
4.4.6 Interference Check Sample	4-11
4.4.7 Method Blank	4-12
4.4.8 Ambient Blank	4-12
4.4.9 Equipment Blank	4-13
4.4.10 Trip Blank	4-13
4.4.11 Field Duplicates	4-13
4.4.12 Field Replicates	4-14
4.5 Quality Control Procedures	4-14
4.5.1 Holding Time Compliance	4-14
4.5.2 Confirmation	4-15
4.5.3 Control Charts	4-15
4.5.4 Standard Materials	4-15
4.5.5 Supplies and Consumables	4-16
5.0 Sampling Procedures	5-1
5.1 Field Sampling	5-1
5.1.1 Sample Containers	5-1
5.1.2 Sample Volumes, Container Types, and Preservation Requirements	5-1
5.2 Sample Handling and Custody	5-6
6.0 Screening Analytical Methods	6-1
6.1 Analytical Screening Method Descriptions	6-1
6.1.1 EPA Method SW1020A—Ignitability	6-2
6.1.2 EPA Method SW1110—Corrosivity	6-2
6.1.3 EPA Method SW9040B (Water)/SW9045C (Soil)—pH	6-2
6.1.4 EPA Method SW9050A—Conductance	6-2
6.1.5 EPA Method SW9060—Total Organic Carbon	6-2
6.1.6 EPA Method 160.1—Filterable Residue	6-3
6.1.7 EPA Method 160.2—Nonfilterable Residue	6-3
6.1.8 EPA Method 170.1—Temperature	6-3
6.1.9 EPA Method 180.1—Turbidity	6-3
6.1.10 EPA Method 310.1—Alkalinity	6-4
6.1.11 EPA Method 360.1—Dissolved Oxygen	6-4
6.1.12 ASTM D422—Standard Method for Particle-Size Analysis of Soils	6-4

<b>SECTION</b>	<b>PAGE</b>
6.1.13 ASTM D1498—Oxidation-Reduction Potential	6-4
6.1.14 ASTM D3416—Methane in Soil Gas	6-4
6.1.15 EPA Method SW4020—Screening for Polychlorinated Biphenyls by Immunoassay	6-5
6.1.16 EPA Method SW4030—Screening for Petroleum Hydrocarbons by Immunoassay	6-5
6.1.17 SW-846 (Described in Method SW3550)—Percent Solids	6-5
6.1.18 Real-Time Portable Organic Vapor Analyzers	6-6
6.2 Calibration and QC Procedures for Screening Methods	6-7
7.0 Definitive Data Analytical Methods and Procedures	7-1
7.1 Preparation Methods	7-1
7.1.1 Method SW1311—Toxicity Characteristic Leaching Procedure	7-2
7.1.2 Method 300.3 – Common Anions in Soil	7-3
7.1.3 Method SW3005A— Acid Digestion of Water Samples for Metals Analysis	7-3
7.1.4 Method SW3010A— Acid Digestion of Aqueous Samples and Extracts for Metals Analysis	7-3
7.1.5 Method SW3015— Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis	7-3
7.1.6 Method SW3020A— Acid Digestion of Aqueous Samples and Extracts for Metals Analysis	7-3
7.1.7 Method SW3050B— Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis	7-3
7.1.8 Method SW3051— Microwave Assisted Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis	7-4
7.1.9 Method SW3060A—Alkaline Digestion for Hexavalent Chromium	7-4
7.1.10 Method SW3510C—Separatory Funnel Liquid-Liquid Extraction	7-4
7.1.11 Method SW3520C—Continuous Liquid-Liquid Extraction	7-4
7.1.12 Method SW3535—Solid-Phase Extraction	7-4
7.1.13 Method SW3540C/SW3541—Soxhlet Extraction	7-4
7.1.14 Method SW3545—Pressurized Fluid Extraction	7-5
7.1.15 Method SW3550B—Ultrasonic Extraction	7-5
7.1.16 Method SW3585—Waste Dilution for Volatile Organics	7-5
7.1.17 Method SW5021—Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis	7-5
7.1.18 Method SW5030B—Purge and Trap	7-5

<b>SECTION</b>	<b>PAGE</b>
7.1.19 Method SW5031—Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation	7-6
7.1.20 Method SW5032—Volatile Organic Compounds by Vacuum Distillation	7-6
7.1.21 Method SW5035—Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples	7-6
7.2 Analytical Procedures	7-6
7.2.1 Method SW8011—Ethylene Dibromide	7-9
7.2.2 Method SW8015 (Modified)—Volatile and Extractable Total Petroleum Hydrocarbons	7-14
7.2.3 Method SW8021B—Aromatic and Halogenated Volatile Organics	7-19
7.2.4 Method SW8070A—Nitrosamines	7-27
7.2.5 Method SW8081A—Organochlorine Pesticides	7-31
7.2.6 Method SW8082—Polychlorinated Biphenyls	7-37
7.2.7 Method SW8141A—Organophosphorus Pesticides	7-43
7.2.8 Method SW8151A—Chlorinated Herbicides	7-49
7.2.9 Method SW8260B—Volatile Organics	7-53
7.2.10 Method SW8270C—Semivolatile Organics	7-61
7.2.11 Method SW8290A—Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans	7-70
7.2.12 Method SW8310—Polynuclear Aromatic Hydrocarbons	7-73
7.2.13 Method SW8321—Chlorinated Herbicides by HPLC/MS	7-79
7.2.14 Method SW8330—Explosive Residues	7-84
7.2.15 Method SW6010B—Trace Elements (Metals) by Inductively Coupled Plasma Atomic Emission Spectroscopy for Water and Soil	7-89
7.2.16 Method SW6020—Trace Elements (Metals) by Inductively Coupled Plasma Mass Spectrometry for Water and Soil	7-95
7.2.17 Method SW7041—Graphite Furnace Atomic Absorption (Antimony)	7-101
7.2.18 Method SW7060A—Graphite Furnace Atomic Absorption (Arsenic)	7-106
7.2.19 Method SW7131A—Graphite Furnace Atomic Absorption (Cadmium)	7-111
7.2.20 Method SW7191—Graphite Furnace Atomic Absorption (Chromium)	7-116
7.2.21 Method SW7196A—Hexavalent Chromium (Colorimetric)	7-121

<b>SECTION</b>	<b>PAGE</b>
7.2.22 Method SW7421—Graphite Furnace Atomic Absorption (Lead)	7-124
7.2.23 Method SW7470A/SW7471A—Mercury Manual Cold-Vapor Technique	7-129
7.2.24 Method SW7521—Graphite Furnace Atomic Absorption (Nickel)	7-133
7.2.25 Method SW7740—Graphite Furnace Atomic Absorption (Selenium)	7-138
7.2.26 Method SW7841—Graphite Furnace Atomic Absorption (Thallium)	7-143
7.2.27 Method SW7911—Graphite Furnace Atomic Absorption (Vanadium)	7-148
7.2.28 Method SW9010B/SW9012A—Total Cyanide and Cyanide Amenable to Chlorination	7-153
7.2.29 Method SW9056—Common Anions	7-157
7.2.30 Method 314.0—Perchlorate Anion	7-162
7.2.31 Method RSK-175—Soil Gases(Volatile Organics) in water	7-167
7.2.32 Method TO-14—Volatile Organics in Ambient Air	7-171
 8.0 Data Reduction, Review, Verification, Reporting, Validation, and Recordkeeping	 8-1
8.1 Data Review, Validation, and Reporting Requirements for Screening Data	8-1
8.2 Data Review, Validation, and Reporting Requirements for Definitive Data	8-2
8.3 Quality Assurance Reports	8-8
8.4 ERPIMS Electronic Data Reports	8-8
8.5 Archiving	8-8
8.6 Project Data Flow and Transfer	8-8
8.7 Recordkeeping	8-8
8.8 Hardcopy Data Reports for Screening and Definitive Data	8-9
 9.0 Systems and Performance Audits, Performance Evaluation Programs, Magnetic Tape Audits, and Training	 9-1
9.1 Project Audits	9-1
9.1.1 State/Federal Project Audits	9-1
9.1.2 Technical Systems Audits	9-1
9.1.3 Project-Specific Performance Evaluation Audits	9-2
9.1.4 Magnetic Tape Audits	9-3

<b>SECTION</b>	<b>PAGE</b>
9.1.5 Performance Evaluation Sample Programs	9-3
9.2 Training	9-3
10.0 Preventive Maintenance	10-1
10.1 Maintenance Responsibilities	10-1
10.2 Maintenance Schedules	10-1
10.3 Spare Parts	10-1
10.4 Maintenance Records	10-1
11.0 Corrective Action	11-1
11.1 Corrective Action Report	11-1
11.2 Corrective Action System	11-1
11.2.1 Manual Integration	11-1
12.0 Quality Assurance Reports to Management	12-1

## **LIST OF TABLES**

<b>TABLE</b>		<b>PAGE</b>
4.2.1-1	Statistical Calculations	4-5
5.1.2-1	Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times	5-2
6-1	Screening Analytical Methods	6-1
6.2-1	Summary of Calibration and QC Procedures for Screening Methods	6-8
7.1-1	Extraction and Digestion Procedures	7-2
7.2-1	Analytical Procedures	7-8
7.2.1-1	RL for Method SW8011	7-9
7.2.1-2	QC Acceptance Criteria for Method SW8011	7-9
7.2.1-3	Summary of Calibration and QC Procedures for Method SW8011	7-10
7.2.2-1	RLs for Method SW8015 (Modified)	7-15
7.2.2-2	QC Acceptance Criteria for Method SW8015 (Modified)	7-15
7.2.2-3	Summary of Calibration and QC Procedures for Method SW8015 (Modified)	7-16
7.2.3-1	RLs for Method SW8021B	7-20
7.2.3-2	QC Acceptance Criteria for Method SW8021B	7-22
7.2.3-3	Summary of Calibration and QC Procedures for Method SW8021B	7-24
7.2.4-1	RLs for Method SW8070A	7-27



<b>TABLE</b>	<b>PAGE</b>
7.2.4-2 QC Acceptance Criteria for Method SW8070A	7-27
7.2.4-3 Summary of Calibration and QC Procedures for Method SW8070A	7-28
7.2.5-1 RLs for Method SW8081A	7-30
7.2.5-2 QC Acceptance Criteria for Method SW8081A	7-32
7.2.5-3 Summary of Calibration and QC Procedures for Method SW8081A	7-34
7.2.6-1 RLs for Method SW8082	7-38
7.2.6-2 QC Acceptance Criteria for Method SW8082	7-39
7.2.6-3 Summary of Calibration and QC Procedures for Method SW8082	7-40
7.2.7-1 RLs for Method SW8141A	7-44
7.2.7-2 QC Acceptance Criteria for Method SW8141A	7-45
7.2.7-3 Summary of Calibration and QC Procedures for Method SW8141A	7-46
7.2.8-1 RLs for Method SW8151A	7-49
7.2.8-2 QC Acceptance Criteria for Method SW8151A	7-49
7.2.8-3 Summary of Calibration and QC Procedures for Method SW8151A	7-50
7.2.9-1 RLs for Method SW8260B	7-54
7.2.9-2 QC Acceptance Criteria for Method SW8260B	7-56
7.2.9-3 Summary of Calibration and QC Procedures for Method SW8260B	7-58
7.2.10-1 RLs for Method SW8270C	7-62
7.2.10-2 QC Acceptance Criteria for Method SW8270C	7-74
7.2.10-3 Summary of Calibration and QC Procedures for Method SW8270C	7-67

<b>TABLE</b>	<b>PAGE</b>
7.2.11-1	RLs for Method SW8290
7.2.11-2	Summary of Calibration and QC Procedures for Method SW8290
7.2.12-1	RLs for Method SW8310
7.2.12-2	QC Acceptance Criteria for Method SW8310
7.2.12-3	Summary of Calibration and QC Procedures for Method SW8310
7.2.13-1	RLs for Method SW8321
7.2.13-2	QC Acceptance Criteria for Method SW8321
7.2.13-3	Summary of Calibration and QC Procedures for Method SW8321
7.2.14-1	RLs for Method SW8330
7.2.14-2	QC Acceptance Criteria for Method SW8330
7.2.14-3	Summary of Calibration and QC Procedures for Method SW8330
7.2.15-1	RLs for Method SW6010B
7.2.15-2	QC Acceptance Criteria for Method SW6010B
7.2.15-3	Summary of Calibration and QC Procedures for Method SW6010B
7.2.16-1	RLs for Method SW6020
7.2.16-2	QC Acceptance Criteria for Method SW6020
7.2.16-3	Summary of Calibration and QC Procedures for Method SW6020
7.2.17-1	RLs for Method SW7041

<b>TABLE</b>		<b>PAGE</b>
7.2.17-2	QC Acceptance Criteria for Method SW7041	7-102
7.2.17-3	Summary of Calibration and QC Procedures for Method SW7041	7-103
7.2.18-1	RLs for Method SW7060A	7-107
7.2.18-2	QC Acceptance Criteria for Method SW7060A	7-107
7.2.18-3	Summary of Calibration and QC Procedures for Method SW7060A	7-108
7.2.19-1	RLs for Method SW7131A	7-112
7.2.19-2	QC Acceptance Criteria for Method SW7131A	7-112
7.2.19-3	Summary of Calibration and QC Procedures for Method SW7131A	7-113
7.2.20-1	RLs for Method SW7191	7-117
7.2.20-2	QC Acceptance Criteria for Method SW7191	7-117
7.2.20-3	Summary of Calibration and QC Procedures for Method SW7191	7-118
7.2.21-1	RLs for Method SW7196A	7-121
7.2.21-2	QC Acceptance Criteria for Method SW7196A	7-121
7.2.21-3	Summary of Calibration and QC Procedures for Method SW7196A	7-122
7.2.22-1	RLs for Method SW7421	7-125
7.2.22-2	QC Acceptance Criteria for Method SW7421	7-125
7.2.22-3	Summary of Calibration and QC Procedures for Method SW7421	7-126
7.2.23-1	RLs for Method SW7470A/SW7471A	7-130
7.2.23-2	QC Acceptance Criteria for Method SW7470A/SW7471A	7-130

<b>TABLE</b>	<b>PAGE</b>
7.2.23-3 Summary of Calibration and QC Procedures for Method SW7470A/SW7471A	7-131
7.2.24-1 RLs for Method SW7521	7-134
7.2.24-2 QC Acceptance Criteria for Method SW7521	7-134
7.2.24-3 Summary of Calibration and QC Procedures for Method SW7521	7-135
7.2.25-1 RLs for Method SW7740	7-139
7.2.25-2 QC Acceptance Criteria for Method SW7740	7-139
7.2.25-3 Summary of Calibration and QC Procedures for Method SW7740	7-140
7.2.26-1 RLs for Method SW7841	7-144
7.2.26-2 QC Acceptance Criteria for Method SW7841	7-144
7.2.26-3 Summary of Calibration and QC Procedures for Method SW7841	7-145
7.2.27-1 RLs for Method SW7911	7-149
7.2.27-2 QC Acceptance Criteria for Method SW7911	7-149
7.2.27-3 Summary of Calibration and QC Procedures for Method SW7911	7-150
7.2.28-1 RLs for Method SW9010A/SW9012A	7-154
7.2.28-2 QC Acceptance Criteria for Method SW9010A/SW9012A	7-154
7.2.28-3 Summary of Calibration and QC Procedures for Method SW9010A/SW9012A	7-155
7.2.29-1 RLs for Method SW9056	7-158
7.2.29-2 QC Acceptance Criteria for Method SW9056	7-158

<b>TABLE</b>		<b>PAGE</b>
7.2.29-3	Summary of Calibration and QC Procedures for Method SW9056	7-159
7.2.30-1	RLs for Method 314.0	7-163
7.2.30-2	QC Acceptance Criteria for Method 314.0	7-163
7.2.30-3	Summary of Calibration and QC Procedures for Method 314.0	7-164
7.2.31-1	RLs for Method RSK-175	7-168
7.2.31-2	QC Acceptance Criteria for Method RSK-175	7-168
7.2.31-3	Summary of Calibration and QC Procedures for Method RSK-175	7-169
7.2.32-1	RLs for Method TO-14	7-172
7.2.32-2	QC Acceptance Criteria for Method TO-14	7-172
7.2.32-3	Summary of Calibration and QC Procedures for Method TO-14	7-173
8.2-1	Data Qualifiers	8-3
8.2-2	General Flagging Conventions	8-4
8.2-3	Flagging Conventions Specific to Organic Methods	8-5
8.2-4	Flagging Conventions Specific to Inorganic Methods	8-7

## 1.0 INTRODUCTION

The Quality Assurance Project Plan (QAPP) presents in specific terms the policies, organization, functions, and Quality Assurance/Quality Control (QA/QC) requirements designed to achieve the data quality goals described in the approved Sampling and Analysis Plan (SAP) for the project. This detailed QAPP, (1) has been prepared for use by contractors who perform environmental services to ensure the data are scientifically valid and defensible, and (2) establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner. This QAPP and a site specific Field Sampling Plan (FSP) shall constitute, by definition, an AFCEE Sampling and Analysis Plan (SAP).

The National Contingency Plan (NCP) specifies circumstances under which a QAPP is necessary for Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response actions. For cleanup actions at the remedial investigation/feasibility study (RI/FS) stage, the NCP requires lead agents to develop sampling and analysis plans which provide a process for obtaining data of sufficient quality and quantity to satisfy data needs. Such sampling and analysis plans must include a quality assurance project plan “which describes policy, organization, and functional activities and the data quality objectives and measures necessary to achieve adequate data for use in selecting the appropriate remedy.” 40 CFR 300.430 (b)(8)(ii).

The U.S. Environmental Protection Agency (EPA) QA policy requires a QAPP for every monitoring and measurement project mandated or supported by the EPA through regulations, contracts, or other formalized means not currently covered by regulation. Guidelines followed in the preparation of this plan are set out in *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (U.S. EPA, 1983a) and *U.S. EPA Region IX QAPP: Guidance for Preparing QAPPs for Superfund Remedial Projects* (U.S. EPA, 1989). Other documents that have been referenced for this plan include *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final* (U.S. EPA, 1988); *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, Draft Final, EPA QA/R-5* (U.S. EPA, 1993), *Compendium of Superfund Field Operations Methods* (U.S. EPA, 1987a); *Data Quality Objectives Process for Superfund, Interim Final Guidance* (U.S. EPA, 1993); and *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (U.S. EPA SW-846, Third Edition and its first, second and third update).

This QAPP is required reading for all staff participating in the work effort. The QAPP shall be in the possession of the field teams and in the laboratories performing all analytical methods. All contractors and subcontractors shall be required to comply with the procedures documented in this QAPP in order to maintain comparability and representativeness of the data produced.

Controlled distribution of the QAPP shall be implemented by the prime contractor to ensure the current version is being used. A sequential numbering system shall be used to identify controlled copies of the QAPP. Controlled copies shall be provided to applicable Air Force managers, regulatory agencies, remedial project managers, project managers, and QA coordinators. Whenever Air Force revisions are made or addenda added to the QAPP, a document control system shall be put into place to assure (1) all parties holding a controlled copy of the QAPP shall receive the revisions/addenda and (2) outdated material is removed from circulation. The document control system does not preclude making and using copies of the QAPP; however, the holders of controlled copies are responsible for distributing additional material to update any copies within their organizations. The distribution list for controlled copies shall be maintained by the prime contractor.

## **2.0 PROJECT DESCRIPTION**

### **2.1 THE U.S. AIR FORCE INSTALLATION RESTORATION PROGRAM**

The objective of the U.S. Air Force Installation Restoration Project (IRP) is to assess past hazardous waste disposal and spill sites at U.S. Air Force installations and to develop remedial actions consistent with the NCP for sites that pose a threat to human health and welfare or the environment. This section presents information on the program origins, objectives, and organization.

The 1976 Resource Conservation Recovery Act (RCRA) is one of the primary federal laws governing the disposal of hazardous wastes. Sections 6001 and 6003 of RCRA require federal agencies to comply with local and state environmental regulations and provide information to the EPA concerning past disposal practices at federal sites. RCRA Section 3012 requires state agencies to inventory past hazardous waste disposal sites and provide information to the EPA concerning those sites.

In 1980, Congress enacted CERCLA (Superfund). CERCLA outlines the responsibility for identifying and remediating contaminated sites in the United States and its possessions. The CERCLA legislation identifies the EPA as the primary policy and enforcement agency regarding contaminated sites.

The 1986 Superfund Amendments and Reauthorization Act (SARA) extends the requirements of CERCLA and modifies CERCLA with respect to goals for remediation and the steps that lead to the selection of a remedial process. Under SARA, technologies that provide permanent removal or destruction of a contaminant are preferable to action that only contains or isolates the contaminant. SARA also provides for greater interaction with public and state agencies and extends the EPA's role in evaluating health risks associated with contamination. Under SARA, early determination of Applicable or Relevant and Appropriate Requirements (ARARs) is required, and the consideration of potential remediation alternatives is recommended at the initiation of an RI/FS. SARA is the primary legislation governing remedial action at past hazardous waste disposal sites.

Executive Order 12580, adopted in 1987, gave various federal agencies, including the Department of Defense (DoD), the responsibility to act as lead agencies for conducting investigations and implementing remediation efforts when they are the sole or co-contributor to contamination on or off their properties.

To ensure compliance with CERCLA, its regulations, and Executive Order 12580, the DoD developed the IRP, under the Defense Environmental Restoration Program, to identify potentially contaminated sites, investigate these sites, and evaluate and select remedial actions for potentially contaminated facilities. The DoD issued the Defense Environmental Quality Program Policy



Memorandum (DEQPPM) 80-6 regarding the IRP program in June 1980, and implemented the policies outlined in this memorandum in December 1980. The NCP was issued by EPA in 1980 to provide guidance on a process by which (1) contaminant release could be reported, (2) contamination could be identified and quantified, and (3) remedial actions could be selected. The NCP describes the responsibility of federal and state governments and those responsible for contaminant releases.

The DoD formally revised and expanded the existing IRP directives and amplified all previous directives and memoranda concerning the IRP through DEQPPM 81-5, dated 11 December 1981. The memorandum was implemented by a U.S. Air Force message dated 21 January 1982.

The IRP is the DoD's primary mechanism for response actions on U.S. Air Force installations affected by the provisions of SARA. In November 1986, in response to SARA and other EPA interim guidance, the U.S. Air Force modified the IRP to provide for an RI/FS program. The IRP was modified so that RI/FS studies could be conducted as parallel activities rather than serial activities. The program now includes ARAR determinations, identification and screening of technologies, and development of alternatives. The IRP may include multiple field activities and pilot studies prior to a detailed final analysis of alternatives. Over the years, requirements of the IRP have been developed and modified to ensure that DoD compliance with federal laws, such as RCRA, NCP, CERCLA, and SARA, can be met.

## **2.2 PURPOSE AND SCOPE**

The purpose, scope, and use of this work effort shall be briefly discussed in Section 2.2 of the FSP.

## **2.3 PROJECT BACKGROUND**

A project background description, including (1) the locations of sites at the base or facility, (2) a summary of the contamination history at each site and (3) the findings from previous investigations shall be included in Section 2.3 and Section 2.4 of the FSP.

## **2.4 PROJECT SCOPE AND OBJECTIVES**

A summary of the objectives and the proposed work for each site shall be included in Section 3.1, Section 3.2 and Section 3.3 of the FSP. The intended use of the data acquired during this project, the data quality objective process and a discussion of how the process specific decision rules were derived shall also be described in Section 3.1 of the FSP.

### **3.0 PROJECT ORGANIZATION AND RESPONSIBILITY**

The project organization and responsibility discussion including (1) a project organizational chart identifying task managers and individuals responsible for performance of the project, (2) a list of names of all key participants, including organization names and telephone numbers for project, field, and laboratory QA officers, (3) a description of the authority given to each key participant with an emphasis on the authority of the key individuals to initiate and approve corrective actions, and (4) the role of regulatory representatives shall be included in Section 4.0 of the FSP.

All contractors and subcontractors shall be identified and the scope of their performance in the project shall be clearly defined. Subcontractors proposed to provide backup services shall be identified. An organizational chart, a list of key personnel, and the previously described descriptive text shall be included for each subcontractor in Section 4.1 of the FSP.

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## **4.0 QUALITY PROGRAM AND DATA QUALITY OBJECTIVES**

Data Quality Objectives (DQOs) specify the data type, quality, quantity, and uses needed to make decisions and are the basis for designing data collection activities. The DQOs for the project are specified in the FSP in Section 3.1.

### **4.1 DATA CATEGORIES**

The two general categories of data used by the Air Force Center for Environmental Excellence (AFCEE) are defined as: (1) screening data and (2) definitive data.

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements than are necessary to produce definitive data. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise. Physical test methods, e.g., dissolved oxygen measurements, temperature and pH measurements, moisture content, turbidity, conductance, etc., have been designated by definition as screening methods (see Section 6).

Screening methods shall be confirmed, as required in Section 3.2 of the FSP, by analyses that generate definitive data. Confirmation samples shall be selected to include both detected and nondetected results from the screening method.

Definitive data are generated using rigorous analytical methods (see Section 7), such as approved EPA reference methods. The data can be generated in a mobile or off-site laboratory. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements (Sections 7 and 8). Definitive data are not restricted in their use unless quality problems require data qualification.

### **4.2 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY**

The basis for assessing each of these elements of data quality is discussed in the following subsections. Precision and accuracy QC limits for each method and matrix are identified in Sections 6 and 7.

#### **4.2.1 Precision**

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. *Analytical* precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. AFCEE uses the laboratory control sample (LCS) to determine the precision of the analytical method. If the recoveries of analytes in the LCS are within established control limits, then precision is within limits. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, rather the comparison is between the sample and samples analyzed in previous batches. *Total* precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples shall be analyzed to assess field and analytical precision, and the precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results. The formula for the calculation of precision is provided in Table 4.2.1-1 as RPD. For replicate analyses, the relative standard deviation (RSD) is determined. The formula for the calculation of RSD is provided in Table 4.2.1-1. The required level of precision differs according to the method, and is listed in the accuracy and precision tables in Section 7.

#### **4.2.2 Accuracy**

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semivolatile organic compounds, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of performance evaluation (PE) samples shall also be used to provide additional information for assessing the accuracy of the analytical data being produced.

Both accuracy and precision are calculated for each AFCEE analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is included in Table 4.2.1-1 as percent recovery (%R) from pure and sample matrices. Accuracy requirements are listed for each method in Section 7.

#### **4.2.3 Representativeness**

Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Representativeness shall be achieved through use of the standard field, sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, and sampling locations. Decisions regarding sample/well/ boring locations and numbers and the statistical sampling design are documented in Section 3.3 of the FSP.

#### **4.2.4 Completeness**

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (e.g. by site). Completeness is calculated and reported for each method, matrix and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an "R" flag (see Section 8 for an explanation of flagging criteria). The requirement for completeness is 95 percent for aqueous samples and 90 percent for soil samples. For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of possible results minus the number of possible results not reported.

The formula for calculation of completeness is presented below:

$$\% \text{ completeness} = \frac{\text{number of valid (i.e., non-R flagged) results}}{\text{number of possible results}}$$

#### **4.2.5 Comparability**

Comparability is the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions

encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms shall support the assessment of comparability. Analysis of performance evaluation (PE) samples and reports from audits shall also be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories. Historical comparability shall be achieved through consistent use of methods and documentation procedures throughout the project.

**Table 4.2.1-1 Statistical Calculations**

Statistic	Symbol	Formula	Definition	Uses
Mean	$\bar{X}$	$\frac{\left( \sum_{i=1}^n x_i \right)}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left( \frac{\sum (x_i - \bar{X})^2}{(n-1)} \right)^{1/2}$	Measure of relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	$(S / \bar{X}) \times 100$	Relative standard deviation, adjusts for magnitude of observations	Used to assess precision for replicate results
Percent Difference	%D	$\frac{x_1 - x_2}{x_1} \times 100$	Measure of the difference of 2 observations	Used to assess accuracy
Relative Percent Difference	RPD	$\left( \frac{(x_1 - x_2)}{(x_1 + x_2) / 2} \right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess total and analytical precision of duplicate measurements
Percent Recovery	%R	$\left( \frac{x_{\text{meas}}}{x_{\text{true}}} \right) \times 100$	Recovery of spiked compound in clean matrix	Used to assess accuracy
Percent Recovery	%R	$\left( \frac{\text{value of spiked sample} - \text{value of unspiked sample}}{\text{Value of added spike}} \right) \times 100$	Recovery of spiked compound in sample matrix	Used to assess matrix effects and total precision
Correlation Coefficient	r	see SW8000B section 7.5.3		Evaluation of “goodness of fit” of a regression line
Coefficient of Determination	COD	see SW8000B section 7.5.3		Evaluation of “goodness of fit” of a polynomial equation

x = Observation (concentration)  
n = Number of observations



## **4.3 METHOD DETECTION LIMITS, AFCEE REPORTING LIMITS, AND INSTRUMENT CALIBRATION REQUIREMENTS**

### **4.3.1 Method Detection Limits**

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these MDLs at least once per twelve month period. The laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project (i.e., before project samples are analyzed) and upon request in the format specified in Section 8. Results less than or equal to the MDL shall be reported as the MDL value and flagged with a “U” (see Section 8).

Laboratories participating in this work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the following instructions:

- (1) Estimate the MDL using one of the following:
  - a) the concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5, or
  - b) the concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in reagent water, or
  - c) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).
- (2) Prepare (i.e., extract, digest, etc.) and analyze seven samples of a matrix spike (ASTM Type II water for aqueous methods, Ottawa sand for soil methods, glass beads of 1 mm diameter or smaller for metals) containing the analyte of interest at a concentration three to five times the estimated MDL.
- (3) Determine the variance ( $S^2$ ) for each analyte as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n (x_i - \bar{x})^2 \right]$$

where  $x_i$  = the  $i$ th measurement of the variable  $x$  and  $\bar{x}$  = the average value of  $x$

$$\bar{X} = \frac{1}{n} \sum_{i=1}^n x_i$$

(4) Determine the standard deviation (s) for each analyte as follows:

$$s = (S^2)^{1/2}$$

(5) Determine the MDL for each analyte as follows:

$$\text{MDL} = 3.14(s)$$

(note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

(6) If the spike level used in step 2 is more than 10 times the calculated MDL, repeat the process using a smaller spiking level.

Where multiple instruments are used, the MDL used for reporting purposes shall represent the least sensitive instrument.

#### **4.3.2 Reporting Limits**

The laboratories participating in this work effort shall compare the results of the MDL demonstrations to the reporting limits (RLs) for each method that is listed in Section 7. The MDL may not be more than one-half the corresponding RL. The laboratories shall also verify RLs by including a standard at or below the RL as the lowest point on the calibration curve. All results shall be reported at or above the MDL values, however, for those results falling between the MDL and the RL, an "F" flag shall be applied to the results indicating the variability associated with the result (see Section 8.0). No results shall be reported below the MDL.

#### **4.3.3 Instrument Calibration**

Analytical instruments shall be calibrated in accordance with the analytical methods. All analytes reported shall be present in the initial and continuing calibrations, and these calibrations shall meet the acceptance criteria specified in Section 7. All results reported shall be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and will only give an estimate of the true concentration. For guidance on dilutions, see pages 8-2 and 8-10. For SW6010 and SW6020, results shall be within the working range determined by linear range studies. Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials.

Instrument calibration shall be checked using all of the analytes listed in the QC acceptance criteria table in Section 7 for the method. This applies equally to multiresponse analytes (except

as noted in Section 7). All calibration criteria shall satisfy SW-846 requirements at a minimum. The initial calibration shall be checked at the frequency specified in the method using materials prepared independently of the calibration standards. Multipoint calibrations shall contain the minimum number of calibration points specified in the method with all points used for the calibration being contiguous. If more than the minimum number of standards is analyzed for the initial calibration, all of the standards analyzed shall be included in the initial calibration. The only exception to this rule is a standard that has been statistically determined as being an outlier can be dropped from the calibration, providing the requirement for the minimum number of standards is met. Acceptance criteria for the calibration check are presented in Section 7. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and gas chromatography/mass spectroscopy (GC/MS) methods, when using RFs to determine analyte concentrations, the average RF from the initial five point calibration shall be used. The continuing calibration shall not be used to update the RFs from the initial five point calibration. The continuing calibration verification cannot be used as the laboratory control sample (LCS). In addition, the concentration used for the calibration verification sample shall be at or below the middle of the calibration curve. Finally, the lowest standard used must be at or below the RL for each analyte in the method.

#### **4.4 ELEMENTS OF QUALITY CONTROL**

QC elements relevant to screening data are presented in Section 6.0. This section presents QC requirements relevant to analysis of environmental samples that shall be followed during all analytical activities for fixed-base, mobile, and field laboratories producing definitive data. The purpose of this QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Laboratory QC samples (e.g., blanks and laboratory control samples) shall be included in the preparation batch with the field samples. An AFCEE analytical batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and that are extracted or digested at the same time and with the same lot of reagents. Matrix spikes and matrix spike duplicates count as environmental samples. The term AFCEE analytical batch also extends to cover samples that do not need separate extraction or digestion (e.g., volatile analyses by purge and trap). This AFCEE analytical batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and analyzed sequentially. The identity of each AFCEE analytical batch shall be unambiguously reported with the analyses so that a reviewer can identify

the QC samples and the associated environmental samples. All references to the analytical batch in the following sections and tables in this QAPP refer to the AFCEE analytical batch.

The type of QC samples and the frequency of use of these samples are discussed below and in the method-specific subsections of Section 7.

#### **4.4.1 Laboratory Control Sample**

The laboratory control sample (LCS) is analyte-free water for aqueous analyses or a choice of Ottawa sand, sodium sulfate, or glass beads 1 mm or smaller in diameter for soil spiked with all analytes listed in the QC acceptance criteria table in Section 7 for the method. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. (The midpoint is defined as the median point in the curve, not the middle of the range). The LCS shall be carried through the complete sample preparation and analysis procedure.

The LCS is used to evaluate each AFCEE analytical batch and to determine if the method is in control. The LCS cannot be used as the continuing calibration verification.

One LCS shall be included in every AFCEE analytical batch. If more than one LCS is analyzed in an AFCEE analytical batch, results from all LCSs analyzed shall be reported. A QC failure of an analyte in any of the LCSs shall require appropriate corrective action including qualification of the failed analyte in all of the samples as required.

The performance of the LCS is evaluated against the QC acceptance limits given in the tables in Section 7. Whenever an analyte in an LCS is outside the acceptance limit, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, all samples in the AFCEE analytical batch shall be reanalyzed for the out-of-control analyte(s). When an analyte in an LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action was ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to all affected results.

#### **4.4.2 Matrix Spike/Matrix Spike Duplicate**

A matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all analytes listed in the QC acceptance criteria table in Section 7 for the method. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS and MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Only AFCEE samples shall be used for spiking. The MS/MSD shall be designated on the chain of custody.

The MS/MSD is used to document the bias of a method due to sample matrix. Thus, for soil samples, laboratories may use the same container for the parent sample, the MS sample, and the MSD sample (except for VOAs), if there is enough sample. The prime contractor should select the samples for MS/MSDs. The sample replicates will be generated in the field, to be used by the laboratory to prepare the appropriate MS/MSDs. They are used to document potential matrix effects associated with a site. The MS/MSD results and flags must be associated or related to samples that are collected from the same site from which the MS/MSD set were collected. AFCEE does not use MSs and MSDs to control the analytical process.

A site specific MS/MSD should be specified for each media, e.g., any different soil, water or sediment for each site during each sampling event which should not to exceed 5 working days in one week. Project managers should designate the MS/MSD and determine if they are site specific based on the project requirements. A minimum of one MS and one MSD shall be designated by the project manager for each site and analyzed with every batch of AFCEE samples in a sample delivery group of up to 20 field samples (i.e. collect up to 20 field samples followed by 2 additional samples designated as MS and MSD). More than one MS/MSD pair may be submitted as part of the sample group of environmental samples, however, project managers must coordinate with the laboratory providing analytical services for most cost effective sampling.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables in Section 7. If either the MS or the MSD is outside the QC acceptance limits, the analytes in all related samples shall be qualified according to the data flagging criteria in Sections 7 and 8.

#### **4.4.3 Surrogates**

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples.

Surrogates are used to evaluate accuracy, method performance, and extraction efficiency.

Surrogates shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.

Whenever a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, reprepare and reanalyze the sample. If corrective actions are not performed or are

ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to the sample results.

#### **4.4.4 Internal Standards**

Internal standards (ISs) are measured amounts of certain compounds added after preparation or extraction of a sample. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects.

ISs shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.

When the IS results are outside of the acceptance limits, corrective actions shall be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to the sample results.

#### **4.4.5 Retention Time Windows**

Retention time windows are used in GC and high performance liquid chromatography (HPLC) analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846 Method 8000B.

When the retention time is outside of the acceptance limits, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze all samples analyzed since the last acceptable retention time check. If corrective actions are not performed, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to the sample results.

#### **4.4.6 Interference Check Sample**

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations.

The ICS is used to verify background and interelement correction factors.

The ICS is run at the beginning and end of each run sequence.

When the interference check sample results are outside of the acceptance limits stated in the method, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICS. If the ICS result is acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to all affected results.

#### **4.4.7 Method Blank**

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank shall be carried through the complete sample preparation and analytical procedure.

The method blank is used to document contamination resulting from the analytical process.

A method blank shall be included in every AFCEE analytical batch.

The presence of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples containing the analyte(s) found in the method blank above the RL shall be reprepared and reanalyzed. No analytical data shall be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to the sample results.

#### **4.4.8 Ambient Blank**

The ambient blank consists of ASTM Type II reagent grade water poured into a volatile organic compound (VOC) sample vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Ambient blanks are used to assess the potential introduction of contaminants from ambient sources (e.g., active runways, engine test cells, gasoline motors in operation, etc.) to the samples during sample collection.

The frequency of collection for ambient blanks is specified in Section 3.2 of the FSP. Ambient blanks shall be collected downwind of possible VOC sources.

#### **4.4.9 Equipment Blank**

An equipment blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures.

The frequency of collection for equipment blanks is specified in Section 3.2 of the FSP. Equipment blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank the appropriate validation flag, as described in Section 8, shall be applied to all sample results from samples collected with the affected equipment.

#### **4.4.10 Trip Blank**

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis of VOCs shall contain one trip blank. For methanol preserved soil samples being analyzed for GRO or VOC, a methanol blank shall be utilized.

When an analyte is detected in the trip blank the appropriate validation flag, as described in Section 8, shall be applied to all sample results from samples in the cooler with the affected trip blank.

#### **4.4.11 Field Duplicates**



A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned a unique identification number in the field. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest.

The frequency of collection for field duplicates is specified in Section 3.2 of the FSP.

#### **4.4.12 Field Replicates**

A field replicate sample, also called a split, is a single sample divided into two equal parts for analysis. The sample containers are assigned a unique identification number in the field. Specific locations are designated for collection of field replicate samples prior to the beginning of sample collection.

Replicate sample results are used to assess precision. The frequency of collection for field replicates is specified in Section 3.2 of the FSP.

### **4.5 QUALITY CONTROL PROCEDURES**

#### **4.5.1 Holding Time Compliance**

All sample preparation and analysis shall be completed within the method-required holding times. The holding time for a sample begins at the time of sample collection. Some methods have more than one holding time requirement (e.g., methods SW8081A, SW8270C, etc.). The preparation holding time is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the applicable method, prior to any necessary extract cleanup and/or volume reduction procedures. If no preparation (e.g., extraction) is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation prior to analysis, the analysis

holding time is calculated from the time of preparation completion to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses.

If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.

#### **4.5.2 Confirmation**

Quantitative confirmation of results at or above the RL for samples analyzed by GC or HPLC shall be required, unless otherwise specified for the method in Section 7, and shall be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector will be used. The result from the primary column/detector is the result that shall be reported. If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.

#### **4.5.3 Control Charts**

Control charts are used to track the performance of laboratory control sample recoveries over time. All analytes spiked into the LCS should be tracked via control charts. These charts are useful in identifying trends and problems in an analytical method. Updating these charts on an annual basis and reviewing them on a quarterly basis for possible trends that could compromise data quality is recommended. These charts can also be used to benchmark a laboratory's performance against AFCEE requirements to determine possible areas to look for improvement.

#### **4.5.4 Standard Materials**

Standard materials, including second source materials, used in calibration and to prepare samples shall be traceable to National Institute Standards and Technology (NIST), EPA, American Association of Laboratory Accreditation (A2LA) or other equivalent AFCEE approved source, if available. If an NIST, EPA or A2LA standard material is not available, the standard material proposed for use shall be included in an addendum to the SAP and approved before use. The standard materials shall be current, and the following expiration policy shall be followed: The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable

analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory shall label standard and QC materials with expiration dates.

A second source standard is used to independently confirm initial calibration. A second source standard is a standard purchased from a different vendor than the vendor supplying the material used in the initial calibration standards. The second source material can be used for the continuing calibration standards or for the LCS (but shall be used for one of the two). Two different lot numbers from the same vendor do not constitute a second source.

#### **4.5.5 Supplies and Consumables**

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents shall be monitored by analysis of LCSs. An inventory and storage system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.

## **5.0 SAMPLING PROCEDURES**

### **5.1 FIELD SAMPLING**

The field sampling procedures for collecting samples and sampling methods shall be included in Section 6.0 of the FSP.

#### **5.1.1 Sample Containers**

Sample containers are purchased precleaned and treated according to EPA specifications for the methods. Sampling containers that are reused are decontaminated between uses by the EPA-recommended procedures (i.e., EPA 540/R-93/051). Containers are stored in clean areas to prevent exposure to fuels, solvents, and other contaminants. Amber glass bottles are used routinely where glass containers are specified in the sampling protocol.

#### **5.1.2 Sample Volumes, Container Types, and Preservation Requirements**

Sample volumes, container types, and preservation requirements for the analytical methods performed on AFCEE samples are listed in Table 5.1.2-1. The required sample volumes, container types, and preservation requirements for analytical methods proposed for project work not listed in Table 5.1.2-1 shall be included in an addendum to the FSP and approved by AFCEE before use.

**Table 5.1.2-1. Requirements for Containers, Preservation Techniques,  
Sample Volumes, and Holding Times**

Name	Analytical Methods	Container <sup>a</sup>	Preservation <sup>b,c</sup>	Minimum Sample Container Size	Maximum Holding Time
Alkalinity	E310.1	P, G	4°C	50 mL	14 days
Common anions	SW9056	P, G	None required	50 mL	28 days for Br <sup>-</sup> , F <sup>-</sup> , Cl <sup>-</sup> , and SO <sub>4</sub> <sup>-2</sup> ; 48 hours for NO <sub>3</sub> <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> and PO <sub>4</sub> <sup>-3</sup>
Perchlorate	314.0	P, G	None required	50 mL	28 days
Cyanide, total and amenable to chlorination	SW9010B SW9012A	P, G, T	4°C; NaOH to pH > 12, 0.6 g ascorbic acid	500 mL or 4 ounces	14 days (water and soil)
Filterable residue	E160.1	P, G	4°C	100 mL	7 days
Nonfilterable residue	E160.2	P, G	4°C	100 mL	7 days
Hydrogen ion (pH) (W, S)	SW9040B/ SW9045C	P, G	None required	N/A	Analyze immediately <sup>d</sup>
Nitrogen, nitrate+nitrite	E353.1	P, G	4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	500 mL	28 days
Conductance	SW9050A	P, G	None required	N/A	Analyze immediately <sup>d</sup>
Temperature	E170.1	P, G	None required	N/A	Analyze immediately <sup>d</sup>
Dissolved oxygen	E360.1	G	None required	500 mL	Analyze immediately <sup>d</sup>
Turbidity	E180.1	P, G	4°C	N/A	48 hours
Total organic carbon	SW9060	P, G, T	4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH < 2	500 mL	28 days
Chromium (VI)	SW7196A	P, G, T	4°C	500 mL or 8 ounces	24 hours (water); 30 days until extraction and 4 days after extraction (soil)
Mercury	SW7470A SW7471A	P, G, T	HNO <sub>3</sub> to pH < 2, 4°C	500 mL or 8 ounces	28 days (water and soil)
Metals (except chromium (VI) and mercury)	SW6010B SW6020 and SW-846 AA methods	P, G, T	HNO <sub>3</sub> to pH < 2, 4°C	500 mL or 8 ounces	180 days (water and soil)

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).

b. No pH adjustment for soil.

c. ~~e.~~ Preservation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is only required when residual chlorine is present.

d. *Measurement should be performed on site.*

**Table 5.1.2-1. Continued**

<b>Name</b>	<b>Analytical Methods</b>	<b>Container<sup>a</sup></b>	<b>Preservation<sup>b,c</sup></b>	<b>Minimum Sample Container Size</b>	<b>Maximum Holding Time</b>
Total petroleum hydrocarbons (TPH)-volatile	SW8015 (modified)	G, Teflon-lined septum, T	4°C, HCl to pH < 2	2 x 40 mL or 4 ounces	14 days (water and soil); 7 days if unpreserved by acid
Total petroleum hydrocarbons (TPH)-extractable	SW8015 (modified)	G, amber, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Aromatic and Halogenated volatiles	SW8021B	G, Teflon-lined septum, T	4°C, HCl to pH < 2, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	2 x 40 mL or 4 ounces	14 days (water and soil); 7 days if unpreserved by acid
Nitrosamines	SW8070A	G, Teflon-lined cap, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Chlorinated herbicides	SW8151A / <a href="#">SW8321</a>	G, Teflon-lined cap, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).

b. No pH adjustment for soil.

c. Preservation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is only required when residual chlorine is present.

**Table 5.1.2-1. Continued**

Name	Analytical Methods	Container <sup>a</sup>	Preservation <sup>b,c</sup>	Minimum Sample Container Size	Maximum Holding Time
Organochlorine pesticides	SW8081A	G, Teflon-lined cap, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Polychlorinated biphenyls (PCBs)	SW8082	G, Teflon-lined cap, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Organophosphorus pesticides/ compounds	SW8141A	G, Teflon-lined cap, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Semivolatile organics	SW8270C	G, Teflon-lined cap, T	4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Methane / Soil Gas	RSK-175	G, Teflon-lined septum, T	4°C	2 x 40 mL or 4 ounces	28 days
Volatile organics	SW8260B	G, Teflon-lined septum, T	4°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , (HCl to pH < 2 for volatile aromatics) <sup>b</sup> <i>(in addition, for soil, freezing to -10°C or methanol )</i>	2 x 40 mL or 4 ounces	14 days (water and soil); 7 days <i>for water</i> if unpreserved by acid, 48 hrs for unpreserved soil

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).

b. No pH adjustment for soil.

c. Preservation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is only required when residual chlorine is present.

**Table 5.1.2-1. Concluded**

<b>Name</b>	<b>Analytical Methods</b>	<b>Container<sup>a</sup></b>	<b>Preservation<sup>b,c</sup></b>	<b>Minimum Sample Container Size</b>	<b>Maximum Holding Time</b>
Polynuclear aromatic hydrocarbons (PAHs)	SW8310	G, Teflon-lined cap, T	4°C, store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Dioxins and furans	SW8280A SW8290	G, Teflon-lined cap, T	4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (kept dark)	1 liter or 8 ounces	30 days until extraction and 45 days after extraction (water and soil)
Ethylene dibromide (EDB)	SW8011	G, Teflon-lined cap, T	4°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	2 x 40 mL	28 days (water)
Explosive residues	SW8330	P, G, T	Cool, 4°C	1 liter or 8 ounces	7 days to extraction (water); 14 days to extraction (soil); analyze-within 40 days after extraction
TCLP	SW1311	G, Teflon-lined cap, T	Cool, 4°C	1 liter or 8 ounces	14 days to TCLP extraction and 14 days after extraction (volatiles); 14 days to TCLP extraction, 7 days to prep extraction and 40 days after prep extraction (semivolatiles); 28 days to TCLP extraction and 28 days after extraction (mercury); 180 days to TCLP extraction and 180 days after extraction (metals)
Volatile Organics	TO-14	SUMMA <sup>®</sup> canister	none		14 days

- a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).  
b. No pH adjustment for soil.  
c. Preservation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is only required when residual chlorine is present.



## **5.2 SAMPLE HANDLING AND CUSTODY**

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in field and laboratory records.

The contractor shall maintain chain-of-custody records for all field and field Quality Control (QC) samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up or, (4) it is in a designated secure area.

The following information concerning the sample shall be documented on the AFCEE chain of custody (COC) form (as illustrated in Section 8):

- Unique sample identification for each [container](#)
- Date and time of sample collection
- Source of sample (including name, location, and sample type)
- Designation of MS/MSD
- Preservative used
- Analyses required
- Name of collector(s)
- Pertinent field data (pH, temperature, etc.)
- Serial numbers of custody seals and transportation cases (if used)
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories
- Bill of lading or transporter tracking number (if applicable)

All samples shall be uniquely identified, labeled, and documented in the field at the time of collection in accordance with (IAW) Section 6.2 of the FSP.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples shall be packed in ice or chemical refrigerant to keep them cool during collection and transportation. During transit, it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. A temperature blank (a volatile organics compounds sampling vial filled with tap water) shall be included in every cooler and used to determine the internal temperature of the cooler upon receipt of the cooler at the laboratory. If the temperature of the samples upon receipt exceeds the temperature requirements, the exceedance shall be documented in laboratory records

and discussed with AFCEE. The decision regarding the potentially affected samples shall also be documented.

Once the samples reach the laboratory, they shall be checked against information on the COC form for anomalies. For the safety of the personnel involved, coolers containing AFCEE samples shall be opened in a hood in case there has been any breakage of container of potentially contaminated sample material. The condition, temperature, and appropriate preservation of samples shall be checked and documented on the COC form. Checking an aliquot of the sample using pH paper is an acceptable procedure except for VOCs where an additional sample is required to check preservation. The occurrence of any anomalies in the received samples and their resolution shall be documented in laboratory records. All sample information shall then be entered into a tracking system, and unique analytical sample identifiers shall be assigned. A copy of this information shall be reviewed by the laboratory for accuracy. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times for methods required routinely for AFCEE work are specified in Table 5.1.2-1.

**Samples not preserved or analyzed in accordance with these requirements shall be resampled and analyzed, at no additional cost to AFCEE.** Subcontracted analyses shall be documented with the AFCEE COC form. Procedures ensuring internal laboratory COC shall also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample shall be communicated to the analysts. Analytical batches shall be created, and laboratory QC samples shall be introduced into each batch.

While in the laboratory, samples shall be stored in limited-access, temperature-controlled areas. Refrigerators, coolers and freezers shall be monitored for temperature seven days a week. Acceptance criterion for the temperatures of the refrigerators and coolers is  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Acceptance criterion for the temperatures of the freezers shall be less than  $0^{\circ}\text{C}$ . All of the cold storage areas shall be monitored by thermometers that have been calibrated with a NIST-traceable thermometer. As indicated by the findings of the calibration, correction factors shall be applied to each thermometer. Records that include acceptance criteria shall be maintained. Samples for volatile organics determination shall be stored separately from other samples, standards, and sample extracts. Samples shall be stored after analysis until disposed of IAW applicable local, state, and federal regulations. Disposal records shall be maintained by the laboratory. Refrigerators storing AFCEE VOA samples shall contain a blank that shall be analyzed at a minimum of every two weeks.

Standard operating procedures (SOPs) describing sample control and custody shall be maintained by the laboratory.

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## 6.0 SCREENING ANALYTICAL METHODS

The analytical screening methods contained in this section are shown in Table 6-1. This section includes brief descriptions of the methods and QC required for screening procedures commonly used to conduct work efforts. The methods and QC procedures were taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (U.S. EPA SW-846, Third Edition, and its first, second and third update), *Methods for Chemical Analysis of Water and Waste* (U.S. EPA 1979), *ASTM Annual Book of Standards* (1993), and from manufacturers' literature.

**Table 6-1. Screening Analytical Methods**

Method	Parameter
SW846 (3550)	Moisture (as % solids)
SW1020A / SW1010	Ignitability
SW1110	Corrosivity
SW9040B	pH (water)
SW9045C	pH (soil)
SW9050A	Conductance
SW9060	Total organic carbon
E160.1	Filterable residue
E160.2	Nonfilterable residue
E170.1	Temperature
E180.1	Turbidity
E310.1	Alkalinity
E360.1	Dissolved oxygen
Organic Vapor (FID and PID)	Soil gas screening-halogenated, aromatic, and petroleum hydrocarbons
ASTM D422	Particle size
ASTM D1498	Oxidation-reduction potential
ASTM D3416	Methane
SW4020	PCBs by Immunoassay
SW4030	TPH by Immunoassay

## 6.1 ANALYTICAL SCREENING METHOD DESCRIPTIONS

Section 6.1 contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description
- The RL (if applicable)

### **6.1.1 EPA Method SW1020A/SW1010-Ignitability**

Method SW1010 makes use of the Pensky-Martens tester to determine the flash point of liquid samples including those that form surface films and/or contain non-filterable suspended solids.

Method SW1020A makes use of the Setaflash Closed Tester to determine the flash point of liquids that have flash points between 0° and 110°C and viscosities lower than 150 stokes at 25°C. If a sample contains non-filterable suspended solids, use SW1010 (Pensky-Martens Ignitability) instead of method 1020.

### **6.1.2 EPA Method SW1110-Corrosivity**

This test exposes steel to liquid waste to determine the corrosivity of the waste.

### **6.1.3 EPA Method SW9040B (Water)/SW9045C (Soil)-pH**

pH measurements shall be performed for water samples using method SW9040. pH measurements of soil samples are performed using method SW9045C. Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode.

### **6.1.4 EPA Method SW9050A-Conductance**

Standard conductivity meters are used. Temperature is also reported.

### **6.1.5 EPA Method SW9060-Total Organic Carbon**

Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide by either catalytic combustion or wet chemical oxidation. The carbon dioxide formed is then either measured directly by an infrared detector or converted to methane and measured by a flame ionization detector. The amount of carbon dioxide or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

Method	Analyte	Water	
SW9060	Total organic carbon	RL	Unit
		1	mg/L

#### 6.1.6 EPA Method 160.1–Filterable Residue

A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180 °C.

Method	Analyte	Water	
E160.1	Total dissolved solids	RL	Unit
		20	mg/L

#### 6.1.7 EPA Method 160.2–Nonfilterable Residue

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105 °C.

Method	Analyte	Water	
E160.2	Total suspended solids	RL	Unit
		10	mg/L

#### 6.1.8 EPA Method 170.1–Temperature

Temperature measurements are made with a mercury-filled or dial type centigrade thermometer, or a thermistor.

#### 6.1.9 EPA Method 180.1–Turbidity

This method is based on a comparison of the light scattered by the sample under defined conditions with the light intensity scattered by a standard reference suspension. The higher the intensity, the greater the turbidity. Turbidity measurements are made in a nephelometer and are reported in terms of nephelometric turbidity units (NTUs). The working range for the method is from 0–40 NTU. Higher levels of turbidity can be measured by diluting the sample with turbidity-free deionized water.

#### 6.1.10 EPA Method 310.1–Alkalinity

In this method, an unaltered sample is titrated to an end point of pH 4.5 using hydrochloric or sulfuric acid.

Method	Analyte	Water	
		RL	Unit
E310.1	Alkalinity <sup>1</sup>	10	mg/L

<sup>1</sup> alkalinity measured as calcium carbonate equivalence

#### 6.1.11 EPA Method 360.1–Dissolved Oxygen

An instrumental probe, usually dependent upon an electrochemical reaction, is used for determination of dissolved oxygen in water. Under steady-state conditions, the current or potential can be correlated with dissolved oxygen concentrations.

#### 6.1.12 ASTM D422–Standard Method for Particle-Size Analysis of Soils

This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75  $\mu\text{m}$  (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75  $\mu\text{m}$  is determined by a sedimentation process using a hydrometer.

#### 6.1.13 ASTM D1498–Oxidation-Reduction Potential

This method is designed to measure the oxidation-reduction potential (ORP) in water, which is defined as the electromotive force between a noble metal electrode and a reference electrode when immersed in a solution.

#### 6.1.14 ASTM D3416–Methane in Soil Gas

An aliquot of the soil gas sample is introduced into a prechromatographic or stripper column which removes hydrocarbons other than methane and carbon monoxide. Methane and carbon monoxide are passed through a chromatographic column where they are separated. The methane is measured by a flame ionization detector (FID). Quantitation is performed by comparing the sample response to the response of a known concentration of methane.

#### 6.1.15 EPA Method SW4020–Screening for Polychlorinated Biphenyls by Immunoassay

Soil samples are screened for total polychlorinated biphenyls (PCBs) using immunoassay test kits. A mini methanol extraction of the soil sample is performed, and the extract and an enzyme conjugate reagent are added to immobilized antibodies. The enzyme conjugate competes with the PCBs in the sample for binding to immobilized anti-PCB antibodies. The test is interpreted by comparing the response produced by the sample to the response produced by a standard.

#### 6.1.16 EPA Method SW4030–Screening for Petroleum Hydrocarbons by Immunoassay

Soil samples are screened for levels of total petroleum hydrocarbons (TPH) using TPH test kits. A mini extraction of the soil sample is performed, and the extract and an enzyme conjugate reagent are added to immobilized antibodies. The enzyme conjugate competes with hydrocarbons for binding to immobilized anti-hydrocarbon antibodies. The test is interpreted by comparing the response produced by the sample to the response produced by a standard.

#### 6.1.17 SW-846 (Described in Method SW3550)–Percent ~~Solids~~Moisture

Percent solids is determined for solid samples undergoing analysis for inorganic and organic analytes. The sample is weighed, dried, and then reweighed. Percent solids is calculated as:

$$\frac{\text{Dried Weight}}{\text{Initial Weight}} \times 100 = \% \text{ solids}$$

The solid content is used to calculate results for soil samples on a dry weight basis using the calculation presented below:

$$\frac{\text{Result of analysis on a wet weight basis}}{\% \text{ solids} / 100} = \text{Result of analysis on a dry weight basis}$$

All MDLs for solids samples shall be reported on a dry weight basis. Soil sample ~~or sediment~~ results shall be reported on a dry weight basis.



### 6.1.18 Real-Time Portable Organic Vapor Analyzers

Two types of portable analyzers shall be used to perform real-time nonspecific analyses of hydrocarbon vapors. The instruments include an FID (e.g., Foxboro Century OVA) and a photoionization detector (PID) (e.g., HNu® Systems [HNu®] trace gas analyzer) organic vapor monitor. One or more of these instruments may be used at a specific site, depending on the contaminant species of interest. When used together, the instruments provide complementary information because they are sensitive to different types of hydrocarbon vapors.

The portable analyzers shall be used as a screening tool to help determine the optimum locations for the collection of samples. Field data recorded on the COC forms give the laboratory analysts an indication of the approximate concentration of contaminants and aid in calculating dilution factors before analysis. Additionally, the real-time instruments are used to aid in selecting the proper level of personal protective equipment and monitoring air emissions during sampling activities. The comparability of results obtained from the PID and FID instruments can be considered only to be within the variability of this type of screening instrument. Comparability is greatest when the instruments are calibrated with the same standards and operated within similar concentration ranges.

The FID uses the principle of hydrogen flame ionization to detect and measure total hydrocarbon vapors. The FID has a dynamic operating range from 1 ppmv to 10 ppmv or 1 ppmv to 100,000 ppmv, depending on the instrument, and provides a nonspecific response to total hydrocarbons. If concentrations exceed the range of the instrument, a dilution probe shall be attached to the FID to allow elevated vapor concentrations to be measured. The instrument is highly sensitive to compounds such as methane, benzene, and acetone, but is less sensitive to alcohols and halogenated compounds.

During operation, a sample is drawn into the probe and transmitted to the detection chamber by an internal pumping system. Inside the chamber, the sample is exposed to a hydrogen flame that ionizes the organic vapors. As the organic vapors burn, the ions produced are collected on an electrode in the chamber, and a current proportional to the hydrocarbon concentration is generated. This current is measured and displayed on the meter.

The PID uses a photoionization detector to detect and measure total hydrocarbon vapors. The instrument has an operating range of 0-2,000 ppm. During operation, a gas sample is drawn into the probe and past an ultraviolet light source by an internal pumping system. Contaminants in the sample are ionized, producing an instrument response if their ionization potential is equal to or less than the ionizing energy supplied by the lamp. The radiation produces a free electron for each molecule of ionized contaminant, which generates a current directly proportional to the number of ions produced. This current is measured and displayed on the meter. The PID measures the *total* value for all species present with ionization potentials less than or equal to that of the lamp.

## **6.2 CALIBRATION AND QC PROCEDURES FOR SCREENING METHODS**

All screening data shall be flagged with an “S” data qualifier to show the reported data are screening data (see Section 8). The other data qualifiers that shall be used with screening data are also shown in Table 6.2-1 and Section 8. Flagging criteria are applied (except for the “S” flag) when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Table 6.2-1 presents the calibration and QC procedures for each method. These requirements as well as the corrective actions and data flagging criteria are included. In this table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the method-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the data flagging criteria that must be applied in the event that the method-required calibration and QC acceptance criteria are not met.

**Table 6.2-1. Summary of Calibration and QC Procedures for Screening Methods**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Data Flagging Criteria <sup>b</sup>
SW-846 <sup>c</sup>	Moisture	Duplicate sample	1 per 20 samples	% solid RPD $\leq$ 15%	Correct problem, repeat measurement. If still out, flag data	J if RPD >15% and $\leq$ 30%  R if RPD > 30%
SW9045C	pH (soil)	2-point calibration with pH buffers	1 per 10 samples analyzed	$\pm$ 0.05 pH unit	Check with new buffers; if still out, repair meter; repeat calibration check	R
		pH 7 buffer	At each sample location	$\pm$ 0.1 pH unit	Recalibrate	R
		Duplicate sample	10% of field samples	$\pm$ 0.1 pH unit	Correct problem, repeat measurement. If still out, repeat calibration and reanalyze samples	J
SW9050A	Conductance	Calibration with KCl standard	Once per day at beginning of testing	$\pm$ 5%	If calibration is not achieved, check meter, standards, and probe; recalibrate	R
		Field duplicate	10% of field samples	$\pm$ 5%	Correct problem, repeat measurement	J
SW9040B	pH (water)	2-point calibration with pH buffers	Once per day	$\pm$ 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	R
		pH 7 buffer	At each sample location	$\pm$ 0.1 pH units	Correct problem, recalibrate	R
		Field duplicate	10% of field samples	$\pm$ 0.1 pH units	Correct problem, repeat measurement	J
E170.1	Temperature	Field duplicate	10% of field samples	$\pm$ 1.0°C	Correct problem, repeat measurement	J

- a. All corrective actions shall be documented, and the records shall be maintained by the prime contractor.
- b. All screening results shall first be flagged with an “S” and also any other appropriate validation flags identified in the Data Flagging Criteria column of the table. For example “SJ”, “SB”, “SR”.
- c. Described in method SW3550.

**Table 6.2-1. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Data Flagging Criteria <sup>b</sup>
E180.1	Turbidity	Calibration with one formazin standard per instrument range used	Once per day at beginning of testing	± 5 units, 0–100 range ± 0.5 units, 0–0.2 range ± 0.2 units, 0–1 range	If calibration is not achieved, check meter; replace if necessary, recalibrate	R
		Field duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J
None	Organic vapor concentrations (FID and PID)	3 point calibration	Monthly	correlation coefficient ≥ 0.995	Recalibrate; check instrument and replace if necessary	R
		Calibration verification and check	Daily at beginning and end of day	Response ± 20% of expected value	Correct problem, recalibrate	R
SW9060	Total organic carbon	Method blank	Daily or one per batch, whichever is more frequent	< RL	Clean system; reanalyze blank. Repeat until analyte < RL	B
		Field duplicate	10% of field samples	RPD < 20%	Repeat measurement	J
E160.1	Filterable residue	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
E160.2	Nonfilterable residue	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
ASTM D1498	Oxidation-reduction potential	Sensitivity verification	Daily	ORP should decrease when pH is increased	If ORP increases, correct the polarity of electrodes. If ORP still does not decrease, clean electrodes and Repeat procedure	R
		Calibration with one standard	Once per day	Two successive readings ± 10 millivolts	Correct problem, recalibrate	R
		Field duplicate	10% of field samples	± 10 millivolts	Correct problem, repeat measurement	J

- a. All corrective actions shall be documented, and the records shall be maintained by the prime contractor.
- b. All screening results shall first be flagged with an “S” and also any other appropriate validation flags identified in the Data Flagging Criteria column of the table. For example “SJ”, “SB”, “SR”.

**Table 6.2-1. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Data Flagging Criteria <sup>b</sup>
SW1110	Corrosivity	Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
E310.1	Alkalinity	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
E360.1	Dissolved oxygen	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW4020	PCBs by immunoassay	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW4030	Petroleum hydrocarbons by immunoassay	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
ASTM D3416	Methane	Single point calibration	Daily, prior to sample analysis	Delineation from database average within $\pm 20\%$	Recalibrate	R
		Method blank	Daily or one per batch, whichever is more frequent	< RL	Clean system; reanalyze blank and Repeat until all analytes < RL	B
		Duplicate	1 per batch or 10%	RPD $\leq 20\%$	Analyze third aliquot: if still out, flag data	J

- a. All corrective actions shall be documented, and the records shall be maintained by the prime contractor.
- b. All screening results shall first be flagged with an "S" and also any other appropriate validation flags identified in the Data Flagging Criteria column of the table. For example "SJ", "SB", "SR".

## **7.0 DEFINITIVE DATA ANALYTICAL METHODS AND PROCEDURES**

Section 7.1 contains brief descriptions of preparation methods. FSection 7.2 contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description
- A table of RLs
- A table of QC acceptance criteria
- A table of calibration procedures, QC procedures, and data validation guidelines

This information was obtained from the *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (U.S. EPA SW-846, Third Edition, and its first, second and third update); *Guidance for Contract Deliverables (GCD)*, Version 1.1, March 1998. Definitions of terms are given in Section 4.0, and data validation procedures are presented in Section 8.0.

### **7.1 PREPARATION METHODS**

Extraction and digestion procedures for liquid and solid matrices presented in this section are outlined in Table 7.1-1.

**Table 7.1-1. Extraction and Digestion Procedures**

<b>Method</b>	<b>Parameter</b>
EPA 300	Common Anions in Soil
SW1311	Toxicity Characteristic Leaching Procedure
SW3005A	Acid Digestion of Water Samples for Metals Analysis
SW3010A	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3015	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3020A	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3050B	Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis
SW3051	Microwave Assisted Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis
SW3060A	Alkaline Digestion for Hexavalent Chromium
SW3510C	Separatory Funnel Liquid-Liquid Extraction
SW3520C	Continuous Liquid-Liquid Extraction
SW3535	Solid-Phase Extraction
SW3540C/SW3541	Soxhlet Extraction
SW3545	Pressurized Fluid Extraction
SW3550B	Ultrasonic Extraction
SW3585	Waste Dilution for Volatile Organics
SW5021	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
SW5030B	Purge and Trap for Volatile Organic Compounds
SW5031	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
SW5032	Volatile Organic Compounds by Vacuum Distillation
SW5035	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

### 7.1.1 Method SW1311–Toxicity Characteristic Leaching Procedure

Method SW1311 is used to prepare samples for determination of the concentration of organic (semivolatile and volatile) and inorganic constituents that are leachable from waste or other material.

QC is accomplished by preparing a toxicity characteristic leaching procedure (TCLP) blank at a rate of one blank for every 20 extractions conducted in the extraction vessel. Additional extract is prepared so one MS is performed for each waste type (samples of similar waste types shall be batched together). One MS must be analyzed in each AFCEE analytical batch. These QA measures are in accordance with the requirements of EPA method SW1311, Section 8.0.

### **7.1.2 Method 300.0-Common Anions in Soil**

Section 11.7 describes an extraction procedure for common anions in a solid matrix. A 10 to 1 water to solid mixture is mixed and filtered prior to analysis.

### **7.1.3 Method SW3005A–Acid Digestion of Water Samples for Metals Analysis**

This method is an acid digestion procedure used to prepare water samples for metals analysis. The digested samples are analyzed for total recoverable and dissolved metals determination by inductively coupled plasma (ICP).

For analysis of total recoverable metals, the entire sample is acidified at collection time. For analysis of dissolved metals, upon collection the samples are filtered then acidified.

### **7.1.4 Method SW3010A–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis**

Method SW3010A prepares aqueous or waste samples for total metals determination by ICP. The samples are vigorously digested with acid and then diluted.

### **7.1.5 Method SW3015–Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis**

This method is used to prepare aqueous or waste samples that contain suspended solids, for total metals determination by graphite furnace atomic absorption spectroscopy (GFAA) or ICP. The samples are digested with acid and heated in a microwave.

### **7.1.6 Method SW3020A–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis**

Method SW3020A prepares aqueous or waste samples for total metals determination by GFAA or ICP. The samples are vigorously digested with acid and then diluted.

### **7.1.7 Method SW3050B–Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis**

Method SW3050B is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by ICP or, for some metals, by GFAA. A sample is digested then refluxed with



acid. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

#### **7.1.8 Method SW3051–Microwave Assisted Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis**

Method SW3051 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by GFAA or ICP. The samples are digested with acid and heated in a microwave. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

#### **7.1.9 Method SW3060A–Alkaline Digestion for Hexavalent Chromium**

Method SW3060A is applicable to the preparation of sediment, sludge, and soil samples for analysis of hexavalent chromium by UV-VIS spectrophotometry. The samples are digested with sodium hydroxide.

#### **7.1.10 Method SW3510C-Separatory Funnel Liquid-Liquid Extraction**

Method SW3510C is designed to quantitatively extract nonvolatile and SVOCs from liquid samples using standard separatory funnel techniques. The sample and the extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method used to analyze the extract.

#### **7.1.11 Method SW3520C-Continuous Liquid-Liquid Extraction**

Method SW3520C is a procedure for isolating organic compounds from aqueous samples and is designed for extraction solvents with greater density than the sample.

#### **7.1.12 Method SW3535A-Solid-Phase Extraction**

Method SW3535A is a procedure for isolating organic compounds from aqueous samples using solid-phase extraction media.

#### **7.1.13 Method SW3540C/SW3541-Soxhlet Extraction**

Method SW3540C is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. Method SW3541 is an automated Soxhlet extraction. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

#### **7.1.14 Method SW3545-Pressurized Fluid Extraction**

Method SW3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, sediments, sludges, and waste solids using elevated temperature and pressure.

#### **7.1.15 Method SW3550B-Ultrasonic Extraction**

Method SW3550B is a procedure for extracting nonvolatile and SVOCs from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

#### **7.1.16 Method SW3585-Waste Dilution for Volatile Organics**

Method SW3585 is a procedure describing a solvent dilution of a non-aqueous waste sample prior to direct injection analysis.

#### **7.1.17 Method SW5021-Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis**

Method SW5021 is a general purpose method for the preparation of VOCs in soils, sediments and solid wastes by GC or GC/MS analysis.

#### **7.1.18 Method SW5030B-Purge and Trap for Volatile Organic Compounds**

Method SW5030B describes sample preparation and extraction for the analysis of VOCs. This method is applicable to aqueous samples and soil / sediment extracts.

An inert gas is then bubbled through the sample solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a GC column.

#### **7.1.19 Method SW5031-Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation**

Method SW5031 is a method for separating nonpurgeable water-soluble and VOCs in aqueous or leachates from solid matrices using azeotropic distillation.

#### **7.2.20 Method SW5032-Volatile Organic Compounds by Vacuum Distillation**

Method SW5032 is a method used to determine volatile organic compounds from a variety of matrices using vacuum distillation.

#### **7.1.21 Method SW5035-Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples**

Method SW5035 describes sample preparation and extraction for the analysis of VOCs in solid matrices. The method involves a heated purge of volatile components followed by analysis on a GC or GC/MS. Several sample preservation options are given in the method. Analyzing the sample unpreserved within the prescribed 48 hour holding time is the preferred option. If this is not possible, an appropriate preservation option must be chosen. For low-level VOC analysis, the preferred preservation is freezing with a 14 day holding time.

### **7.2 ANALYTICAL PROCEDURES**

The analytical procedures presented in this section are outlined in Table 7.2-1.

A brief description and three tables for each method are included in the following subsections. The first table presents the RLs for each analyte in the method. The RLs are presented for both soil and water matrices. The analytes included in these tables are not all inclusive lists. The specific lists of analytes for each method should be determined by regulatory requirements and site specific information. The second table presents the acceptance criteria for the accuracy of spiked analyte and surrogate recoveries. This table also presents the acceptance criteria for the precision of matrix, field, and laboratory duplicate recoveries. The third table presents the calibration and QC procedures for each method. Corrective actions and data flagging criteria are also included in this table.

In the third table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the method-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the

data flagging criteria that shall be applied in the event that the method-required calibration and QC acceptance criteria are not met.

**Table 7.2-1. Analytical Procedures**

Analytical Method	Parameter	Analytical Methods
8011	Ethylene dibromide (EDB) (water)	8011, 5030B
8015 (modified)	TPH volatile and extractable (water and soil)	(volatiles) 5030B, 5031, 5035 (extractables) 3510C, 3520C, 3545C, 3541, 3545, 3550B
8021B	Aromatic and halogenated volatile organics (water and soil)	3585, 5021, 5030B, 5035
8070A	Nitrosamines (water and soil)	3510C, 3520C, 3540C, 3541, 3545, 3550B
8081A	Organochlorine pesticides (water and soil)	3510C, 3520C, 3540C, 3541, 3545, 3550B, 3535A
8082	PCBs (water and soil)	3510C, 3520C, 3540C, 3541, 3535A
8141A	Organophosphorus compounds (water and soil)	3510C, 3520C, 3540C, 3541, 3550B, 3535A
8151A	Chlorinated herbicides (water and soil)	3510C, 3520C, 3540C, 3541, 3550B
8260B	Volatile organics (water and soil)	3585, 5021, 5030B, 5031, 5032, 5035
8270C	Semivolatile organics (water and soil)	3510C, 3520C, 3540C, 3541, 3545, 3550B, 3535A
8290	Dioxins and furans (water and soil)	(see analytical method)
8310	Polynuclear aromatic hydrocarbons (PAHs) (water and soil)	3510C, 3520C, 3540C, 3541, 3550B
8321	Chlorinated herbicides by HPLC-MS (water and soil)	3510C, 3520C, 3540C, 3541, 3550B
8330	Explosive residues (water and soil)	3510C, 3520C, 3535A, 3540C, 3541, 3550B
6010B	Trace metals by ICPEs (water and soil)	3005A, 3010A, 3015, 3050B, 3051
6020	Trace metals by ICP-MS (water and soil)	3005A, 3010A, 3015, 3050B, 3051
7041	Antimony (water and soil)	(see analytical method), 3005A
7060A	Arsenic (water and soil)	(see analytical method), 3050B
7131A	Cadmium (water and soil)	3015, 3020A, 3050B, 3051
7191	Chromium (water and soil)	3015, 3020A, 3050B, 3051
7196A	Hexavalent chromium	3060A
7421	Lead (water and soil)	3015, 3020A, 3050B, 3051
7470A	Mercury (water)	(see analytical method)
7471A	Mercury (soil)	(see analytical method)
7521	Nickel (water and soil)	3015, 3020A, 3050B, 3051
7740	Selenium (water and soil)	(see analytical method), 3050B
7841	Thallium (water and soil)	3015, 3020A, 3050B, 3051
7911	Vanadium (water and soil)	3015, 3020A, 3050B, 3051
9010B	Cyanide (water)	(see analytical method)
9012A	Cyanide (water)	(see analytical method)
9056A	Common anions (water)	(see analytical method)
9056A	Common anions (soil)	As per EPA 300.0
314.0	Perchlorate anion	(see analytical method)
RSK-175	Soil gasses in water	N/A
TO-14	Volatile Organics in Ambient Air	N/A



## 7.2.1 Method SW8011-Ethylene Dibromide

Ethylene dibromide (EDB) in water is analyzed using method SW8011. The sample is extracted with hexane. The extract is injected into a GC with a linearized electron capture detector for separation and analysis. The RL is presented in Table 7.2.1-1.

This method provides for the use of a second GC column of dissimilar phase to resolve compounds of interest from interferences that may occur. When second-column analysis is performed, retention times for the analyte must match those established for each column. Otherwise, the chromatographic peaks are considered interferences, and the analyte is not considered to be present in the sample. Requirements for confirmation of the analyte are described in Section 4.5.2. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.1-2 and 7.2.1-3.

**Table 7.2.1-1. RL for Method SW8011**

Parameter/Method	Analyte	Water	
		RL	Unit
SW8011	EDB	0.02	µg/L

**Table 7.2.1-2. QC Acceptance Criteria for Method SW8011**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW8011	EDB	80-120	≤ 20
	<i>Surrogate:</i> 1,2-Dibromopropane	70-120	
	or 1,2-Dichloropropane	70-120	

**Table 7.2.1-3. Summary of Calibration and QC Procedures for Method SW8011**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8011	EDB	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	Apply R to the result for EDB for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to the result for EDB for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	$\pm 3$ times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to the result for EDB in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to the result for EDB for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to the result for EDB in all samples since the last acceptable calibration verification
		Calibration blank	Once per daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration	Apply B to all results for the specific



			verification		blank and all samples associated with blank	analyte in all samples associated with the blank
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**Table 7.2.1-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8011	EDB	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.1-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to the EDB result for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the result for EDB in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.1-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For EDB in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.1-2	Correct problem then reextract and analyze sample	<p>For the samples; if the %R &gt; UCL for any surrogate, apply J to all positive results</p> <p>if the %R &lt; LCL for any surrogate, apply J to all positive results, apply R to all non-detects</p> <p>If any surrogate recovery is &lt; 10%, apply R to all results</p>
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**Table 7.2.1-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8011	EDB	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.1-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.1-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### **7.2.2 Method SW8015 (Modified)-Volatile and Extractable Total Petroleum Hydrocarbons**

Volatile petroleum hydrocarbon components, such as gasoline, jet fuel, and other low molecular weight petroleum products, are analyzed by the direct purge and trap technique described in method SW5030B followed by a modified approach to method SW8015. Extractable TPH components are analyzed by extraction followed by GC analysis.

For volatile TPH, the sample is placed in the purge and trap sparge vessel and analysis is conducted using a GC equipped with a FID.

Extractable TPH components, such as kerosene, diesel, motor oil, and other high molecular weight extractable petroleum products, are typically prepared by method SW3520C or SW3510C for water-based matrices or by method SW3550B for soil/sludge matrices. Other extraction options are listed in table 7.2.1. After the sample is extracted, analysis is accomplished on a GC equipped with a capillary or megabore column and a FID. RLs for volatile TPH and extractable TPH are provided in Table 7.2.2-1.

Identification and quantitation of TPH components require more analytical judgment than other GC methods. The TPH chromatograms consist of groups of peaks that fall within a noted carbon retention time range (i.e., number of carbon atoms in the molecule). Standard fuel components are used to calibrate the instruments. The total petroleum hydrocarbons results are reported in mg/kg or mg/L based on quantitation of the total area count for the gasoline range organics (i.e., C6-C10) or the diesel range organics (i.e., C10-C28). The retention time window shall be set such that the window encompasses only the C6 through C28 range of organics. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.2-2 and 7.2.2-3. Second column confirmation is not required.

**Table 7.2.2-1. RLs for Method SW8015 (Modified)**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Petroleum Hydrocarbons SW8015 (Mod)	Gasoline	0.1	mg/L	1.0	mg/kg
	Diesel	1.0	mg/L	10.0	mg/kg
	Jet Fuel	1.0	mg/L	10.0	mg/kg

**Table 7.2.2-2. QC Acceptance Criteria for Method SW8015 (Modified)**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8015 (Modified) GRO	TPH–Gasoline	67–136	≤ 30	57–146	≤ 50
	<i>Surrogate:</i> Chlorobenzene	74–138		64–148	
SW8015 (Modified) DRO	TPH–Diesel	61–143	≤ 30	51–153	≤ 50
	TPH–Jet Fuel	61–143	≤ 30	51–153	≤ 50
	<i>Surrogates (choose 2):</i>				
	Octacosane	26–152		25–162	
	Ortho-Terphenyl	57–132		47–142	
	Fluorobenzene	75–125		65–135	
	Tricontane	40–140		30–150	

**Table 7.2.2-3. Summary of Calibration and QC Procedures for Method SW8015 (Modified)**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Continuing calibration verification	Daily, before sample analysis	All concentration levels of GRO within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All concentration levels within ±15% of initial calibration	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.2-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst

**Table 7.2.2-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	Method blank	One per analytical batch	No TPH detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.2-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.2-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results

**Table 7.2.2-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.2-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Retention time window calculated	Each initial calibration	GRO - calculate retention time based on 2-methylpentane and 1,2,4-trimethylbenzene (see 7.4.2 in method) DRO - calculate retention time based on C10 and C28 alkanes (see 7.4.3 in method)	Correct problem then reanalyze all samples analyzed since the last valid retention time check	Apply R to the results from the sample
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.2-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



### **7.2.3 Method SW8021B- Aromatic and Halogenated Volatile Organics**

Aromatic and halogenated volatile organics in water and soil samples are analyzed using method SW8021B. This method is a purge and trap GC method using preparation method SW5030B or SW5035. A temperature program is used in the GC to separate the compounds. Detection is achieved by a PID and an electrolytic conductivity detector (HECD) in series. The RLs for the analytes are presented in Table 7.2.3-1. Requirements for confirmation of analytes are described in Section 4.5.2. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.3-2 and 7.2.3-3.

For analytes detected by both detectors, no further confirmation need be performed. For analytes detected by only one detector, confirmation on another column is required.

**Table 7.2.3-1. RLs for Method SW8021B**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Aromatic and Halogenated Volatile Organics SW8021B	1,1,1,2-Tetrachloroethane	0.50	µg/L	0.01	mg/kg
	1,1,1-TCA	1.0	µg/L	0.01	mg/kg
	1,1,2,2-Tetrachloroethane	0.50	µg/L	0.01	mg/kg
	1,1,2-TCA	0.50	µg/L	0.01	mg/kg
	1,1-DCA	1.0	µg/L	0.01	mg/kg
	1,1-DCE	1.0	µg/L	0.01	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.01	mg/kg
	1,2,3-Trichlorobenzene	1.0	µg/L	0.01	mg/kg
	1,2,3-Trichloropropane	4.0	µg/L	0.01	mg/kg
	1,2,4-Trichlorobenzene	1.0	µg/L	0.01	mg/kg
	1,2,4-Trimethylbenzene	1.0	µg/L	0.01	mg/kg
	1,2-Dibromo-3-chloropropane	30	µg/L	0.03	mg/kg
	1,2-Dibromoethane (EDB)	8.0	µg/L	0.02	mg/kg
	1,2-DCA	0.30	µg/L	0.01	mg/kg
	1,2-DCB	1.0	µg/L	0.01	mg/kg
	1,2-Dichloropropane	1.0	µg/L	0.01	mg/kg
	1,3,5-Trimethylbenzene	1.0	µg/L	0.01	mg/kg
	1,3-DCB	1.0	µg/L	0.01	mg/kg
	1,3-Dichloropropane	0.30	µg/L	0.01	mg/kg
	1,4-DCB	0.50	µg/L	0.01	mg/kg
	2,2-Dichloropropane	1.0	µg/L	0.01	mg/kg
	2-Chlorotoluene	1.0	µg/L	0.01	mg/kg
	4-Chlorotoluene	1.0	µg/L	0.01	mg/kg
	Benzene	0.20	µg/L	0.01	mg/kg
	Bromobenzene	1.0	µg/L	0.01	mg/kg
	Bromochloromethane	1.0	µg/L	0.01	mg/kg
	Bromodichloromethane	0.20	µg/L	0.01	mg/kg
	Bromoform	1.0	µg/L	0.01	mg/kg
	Bromomethane	5.0	µg/L	0.01	mg/kg
	Carbon Tetrachloride	0.10	µg/L	0.01	mg/kg
	Chlorobenzene	0.50	µg/L	0.01	mg/kg
	Chloroethane	1.0	µg/L	0.01	mg/kg
	Chloroform	0.20	µg/L	0.01	mg/kg
	Chloromethane	0.50	µg/L	0.01	mg/kg
	Cis-1,2-DCE	1.0	µg/L	0.01	mg/kg
	Cis-1,3-Dichloropropene	0.50	µg/L	0.01	mg/kg
	Dibromochloromethane	0.50	µg/L	0.01	mg/kg
	Dibromomethane	1.0	µg/L	0.01	mg/kg
	Dichlorodifluoromethane	1.0	µg/L	0.01	mg/kg
	Ethylbenzene	1.0	µg/L	0.01	mg/kg
	Hexachlorobutadiene	0.60	µg/L	0.01	mg/kg
	Isopropylbenzene	1.0	µg/L	0.01	mg/kg
	m,p-Xylene	2.0	µg/L	0.01	mg/kg
	Methylene Chloride	1.0	µg/L	0.01	mg/kg

**Table 7.2.3-1. Concluded**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Aromatic and Halogenated Volatile Organics SW8021B	n-Butylbenzene	1.0	µg/L	0.01	mg/kg
	n-Propylbenzene	1.0	µg/L	0.01	mg/kg
	Naphthalene	1.0	µg/L	0.01	mg/kg
	o-Xylene	1.0	µg/L	0.01	mg/kg
	p-Isopropyltoluene	1.0	µg/L	0.01	mg/kg
	Sec-Butylbenzene	1.0	µg/L	0.01	mg/kg
	Styrene	1.0	µg/L	0.01	mg/kg
	TCE	0.50	µg/L	0.01	mg/kg
	Tert-Butylbenzene	1.0	µg/L	0.01	mg/kg
	Tetrachloroethylene	0.50	µg/L	0.01	mg/kg
	Toluene	1.0	µg/L	0.01	mg/kg
	Trans-1,2-DCE	1.0	µg/L	0.01	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.01	mg/kg
	Trichlorofluoromethane	1.0	µg/L	0.01	mg/kg
	Vinyl Chloride	0.40	µg/L	0.01	mg/kg
	Xylenes, Total	1.0	µg/L	0.01	mg/kg

**Table 7.2.3-2. QC Acceptance Criteria for Method SW8021B**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8021B	1,1,1,2-Tetrachloroethane	75-125	≤ 20	65-125	≤ 30
	1,1,1-TCA	69-134	≤ 20	59-134	≤ 30
	1,1,2,2-Tetrachloroethane	30-166	≤ 20	25-166	≤ 30
	1,1,2-TCA	61-130	≤ 20	51-130	≤ 30
	1,1-DCA	64-127	≤ 20	54-127	≤ 30
	1,1-DCE	53-147	≤ 20	43-147	≤ 30
	1,1-Dichloropropene	65-135	≤ 20	55-145	≤ 30
	1,2,3-Trichlorobenzene	65-135	≤ 20	55-145	≤ 30
	1,2,3-Trichloropropane	75-125	≤ 20	65-125	≤ 30
	1,2,4-Trichlorobenzene	65-135	≤ 20	55-145	≤ 30
	1,2,4-Trimethylbenzene	65-135	≤ 20	55-145	≤ 30
	1,2-Dibromo-3-chloropropane	65-135	≤ 20	55-145	≤ 30
	1,2-Dibromoethane	65-135	≤ 20	55-145	≤ 30
	1,2-DCA	68-137	≤ 20	58-137	≤ 30
	1,2-DCB	61-134	≤ 20	51-134	≤ 30
	1,2-Dichloropropane	73-125	≤ 20	63-125	≤ 30
	1,3,5-Trimethylbenzene	65-135	≤ 20	55-145	≤ 30
	1,3-DCB	63-137	≤ 20	53-137	≤ 30
	1,3-Dichloropropane	65-135	≤ 20	55-145	≤ 30
	1,4-DCB	66-135	≤ 20	56-135	≤ 30
	2,2-Dichloropropane	65-135	≤ 20	55-145	≤ 30
	2-Chlorotoluene	65-135	≤ 20	55-145	≤ 30
	4-Chlorotoluene	65-135	≤ 20	55-145	≤ 30
	Benzene	75-125	≤ 20	65-125	≤ 30
	Bromobenzene	75-125	≤ 20	65-125	≤ 30
	Bromochloromethane	65-135	≤ 20	55-145	≤ 30
	Bromodichloromethane	61-135	≤ 20	51-135	≤ 30
	Bromoform	58-129	≤ 20	48-129	≤ 30
	Bromomethane	68-125	≤ 20	58-125	≤ 30
	Carbon Tetrachloride	69-139	≤ 20	59-139	≤ 30
	Chlorobenzene	75-129	≤ 20	65-129	≤ 30
	Chloroethane	75-130	≤ 20	65-130	≤ 30
	Chloroform	49-133	≤ 20	39-133	≤ 30
	Chloromethane	59-154	≤ 20	49-154	≤ 30
	Cis-1,2-DCE	75-120	≤ 20	65-125	≤ 30
	Cis-1,3-Dichloropropene	75-130	≤ 20	65-130	≤ 30
	Dibromochloromethane	75-131	≤ 20	65-131	≤ 30
	Dibromomethane	65-135	≤ 20	55-145	≤ 30
	Dichlorodifluoromethane	68-125	≤ 20	58-125	≤ 30
	EDB	75-131	≤ 20	65-131	≤ 30
	Ethylbenzene	71-129	≤ 20	61-129	≤ 30
	Hexachlorobutadiene	65-135	≤ 20	55-145	≤ 30

Table 7.2.3-2. Concluded

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8021B	Isopropylbenzene	65-135	≤ 20	55-145	≤ 30
	m,p -Xylene	65-135	≤ 20	55-145	≤ 30
	Methylene Chloride	42-176	≤ 20	32-176	≤ 30
	n-Butylbenzene	65-135	≤ 20	55-145	≤ 30
	n-Propylbenzene	65-135	≤ 20	55-145	≤ 30
	Naphthalene	65-135	≤ 20	55-145	≤ 30
	o-Xylene	65-135	≤ 20	55-145	≤ 30
	p-Isopropyltoluene	65-135	≤ 20	55-145	≤ 30
	Sec-Butylbenzene	65-135	≤ 20	55-145	≤ 30
	Styrene	65-135	≤ 20	55-145	≤ 30
	TCE	75-141	≤ 20	65-141	≤ 30
	Tert-Butylbenzene	65-135	≤ 20	55-145	≤ 30
	Tetrachloroethene	75-142	≤ 20	65-142	≤ 30
	Toluene	70-125	≤ 20	60-125	≤ 30
	Trans-1,2-DCE	75-130	≤ 20	68-130	≤ 30
	Trans-1,3-Dichloropropene	42-156	≤ 20	32-156	≤ 30
	Trichlorofluoromethane	75-130	≤ 20	69-130	≤ 30
	Vinyl Chloride	47-142	≤ 20	37-142	≤ 30
	Xylenes, Total	71-133	≤ 20	61-133	≤ 30
	<i>Surrogates:</i>				
	1,4-Dichlorobutane	35-135		35-135	
	Bromochlorobenzene	37-137		37-137	

**Table 7.2.3-3. Summary of Calibration and QC Procedures for Method SW8021B**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8021B	Aromatic and Halo-genated volatile organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r > 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	$\pm 3$ times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value (for low boiling compounds, see footnote c)	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value (for low boiling compounds, see footnote c)	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.3-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8021B	Halogenated volatile organics	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.3-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.3-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.3-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results

**Table 7.2.3-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8021B	Halogenated volatile organics	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.3-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.3-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
- c. Bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane and vinyl chloride may be within  $\pm 20\%$  of expected value.



#### 7.2.4 Method SW8070A-Nitrosamines

Select nitrosamines in water and soil samples are analyzed using method SW8070A. The sample is extracted and analyzed by gas chromatography. RLs for method SW8070A are presented in Table 7.2.4-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.4-2 and 7.2.4-3.

**Table 7.2.4-1. RLs for Method SW8070A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Nitrosamines SW8070A	N-Nitrosodi-n-propylamine	2.0	µg/L	4.0	mg/kg
	N-Nitrosodimethylamine	0.50	µg/L	1.0	mg/kg
	N-Nitrosodiphenylamine	3.0	µg/L	6.0	mg/kg

**Table 7.2.4-2. QC Acceptance Criteria for Method SW8070A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8070A	N-Nitrosodi-n-propylamine	45–146	≤ 30	35–146	≤ 50
	N-Nitrosodimethylamine	25–125	≤ 30	25–135	≤ 50
	N-Nitrosodiphenylamine	25–139	≤ 30	25–149	≤ 50
	<i>Surrogates<sup>a</sup>:</i>				

- a. Use an analyte and its LCS limit from the method that is not expected to be present in the sample as the surrogate.

**Table 7.2.4-3. Summary of Calibration and QC Procedures for Method SW8070A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8070A	Nitros- amines	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r > 0.995$ for each analyte		
				non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second- source calibration verification	Once per five- point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.4-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8070A	Nitros- amines	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.4-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated analytical blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.4-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.4-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results

**Table 7.2.4-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8070A	Nitros- amines	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.4-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.4-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### **7.2.5 Method SW8081A-Organochlorine Pesticides**

Organochlorine pesticides in water and soil samples are analyzed using method SW8081A. This analytical method involves the extraction of the samples. The pesticides are then separated and quantified by GC using electron capture detection. Reporting limits (RLs) for this method are presented in Table 7.2.5-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.5-2 and 7.2.5-3.

A second-column confirmation is not required for the analysis of toxaphene or chlordane.

**Table 7.2.5-1. RLs for Method SW8081A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Organochlorine Pesticides SW8081A	$\alpha$ -BHC	0.1	$\mu\text{g/L}$	0.004	mg/kg
	$\beta$ -BHC	0.1	$\mu\text{g/L}$	0.004	mg/kg
	$\delta$ -BHC	0.1	$\mu\text{g/L}$	0.004	mg/kg
	$\gamma$ -BHC (Lindane)	0.1	$\mu\text{g/L}$	0.004	mg/kg
	$\alpha$ -Chlordane	0.1	$\mu\text{g/L}$	0.004	mg/kg
	$\gamma$ -Chlordane	0.1	$\mu\text{g/L}$	0.004	mg/kg
	4,4'-DDD	0.1	$\mu\text{g/L}$	0.004	mg/kg
	4,4'-DDE	0.1	$\mu\text{g/L}$	0.004	mg/kg
	4,4'-DDT	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Aldrin	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Dieldrin	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Endosulfan I	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Endosulfan II	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Endosulfan Sulfate	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Endrin	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Endrin Aldehyde	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Heptachlor	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Heptachlor Epoxide	0.1	$\mu\text{g/L}$	0.004	mg/kg
	Methoxychlor	0.5	$\mu\text{g/L}$	0.02	mg/kg
	Toxaphene	1.0	$\mu\text{g/L}$	0.10	mg/kg

**Table 7.2.5-2. QC Acceptance Criteria for Method SW8081A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8081A	α-BHC	60-128	≤ 30	62-125	≤ 50
	β-BHC	66-126	≤ 30	62-127	≤ 50
	δ-BHC	46-136	≤ 30	57-130	≤ 50
	γ-BHC (Lindane)	30-146	≤ 30	59-123	≤ 50
	α-Chlordane	63-123	≤ 30	63-121	≤ 50
	γ-Chlordane	67-120	≤ 30	48-124	≤ 50
	4,4-DDD	50-139	≤ 30	50-139	≤ 50
	4,4-DDE	48-137	≤ 30	68-126	≤ 50
	4,4-DDT	47-138	≤ 30	46-135	≤ 50
	Aldrin	42-138	≤ 30	47-120	≤ 50
	Dieldrin	62-129	≤ 30	67-125	≤ 50
	Endosulfan I	49-120	≤ 30	41-147	≤ 50
	Endosulfan II	42-130	≤ 30	37-141	≤ 50
	Endosulfan Sulfate	54-137	≤ 30	62-135	≤ 50
	Endrin	56-134	≤ 30	61-133	≤ 50
	Endrin Aldehyde	56-137	≤ 30	37-147	≤ 50
	Heptachlor	51-128	≤ 30	51-140	≤ 50
	Heptachlor Epoxide	62-131	≤ 30	66-130	≤ 50
	Methoxychlor	56-150	≤ 30	57-143	≤ 50
	Toxaphene	41-126	≤ 30	31-136	≤ 50
	<i>Surrogates:</i>				
	DCBP	32-135		56-132	
	TCMX	33-138		69-124	

**Table 7.2.5-3. Summary of Calibration and QC Procedures for Method SW8081A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8081A	Organo-chlorine pesticides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification for all analytes	Once per five-point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification



**Table 7.2.5-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8081A	Organo-chlorine pesticides	Breakdown check (Endrin and DDT)	Daily prior to analysis of samples	Degradation ≤15%	Repeat breakdown check	Apply J to all positive DDT, DDE, DDD, endrin, endrin ketone and endrin aldehyde results; apply R to the analytes listed above if minimum frequency is not met
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.5-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.5-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

**Table 7.2.5-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8081A	Organo-chlorine pesticides	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.5-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.5-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation (excluding toxaphene and chlordane)	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.5-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.6 Method SW8082-Polychlorinated Biphenyls (PCBs)**

PCBs in water and soil samples are analyzed using method SW8082. This analytical method involves the extraction of the samples. The PCBs are then separated and quantified by GC using an electron capture detector or electrolytic conductivity detector. Practical quantitation limits (RLs) for this method are presented in Table 7.2.6-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.6-2 and 7.2.6-3.

For analysis of PCBs, the initial five-point calibration and second source calibration verification standards shall, as a minimum contain a mixture of the Aroclors 1016 and 1260. Retention times shall be set during the initial five-point calibration. The, initial and daily calibration verifications may be done using an Aroclor 1016/1260 PCB mixture. Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to validate the linearity of the detector, the single standards of the remaining five Aroclors may be used to determine the response factor for each Aroclor. The concentrations of the individual Aroclor standards should be at or below the middle of the linear range of the detector. If an Aroclor other than 1016 or 1260 is detected (i.e. qualitatively identified above the MDL based on its pattern), report the result for that Aroclor using the response factor from the single Aroclor standard (linear through origin). The LCS and MS/MSD should be spiked using the 1016/1260 mix. A second-column confirmation is not required.

**Table 7.2.6-1. RLs for Method SW8082**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
PCBs	PCB-1016	0.5	µg/L	0.05	mg/kg
	PCB-1221	0.5	µg/L	0.05	mg/kg
	PCB-1232	0.5	µg/L	0.05	mg/kg
	PCB-1242	0.5	µg/L	0.05	mg/kg
	PCB-1248	0.5	µg/L	0.05	mg/kg
	PCB-1254	0.5	µg/L	0.05	mg/kg
	PCB-1260	0.5	µg/L	0.05	mg/kg

**Table 7.2.6-2. QC Acceptance Criteria for Method SW8082**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8082	PCB-1016	40-144	≤ 30	41-138	≤ 50
	PCB-1221	41-136	≤ 30	45-136	≤ 50
	PCB-1232	41-136	≤ 30	45-136	≤ 50
	PCB-1242	39-150	≤ 30	43-150	≤ 50
	PCB-1248	41-136	≤ 30	44-136	≤ 50
	PCB-1254	29-141	≤ 30	41-141	≤ 50
	PCB-1260	45-145	≤ 30	61-131	≤ 50
	1016/1260 Mix	50 - 135	≤ 30	40 - 130	≤ 50
	<i>Surrogate:</i> DCBP	42-133		58-125	

**Table 7.2.6-3. Summary of Calibration and QC Procedures for Method SW8082**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8082	PCBs	Five-point initial calibration <del>for all analytes</del>	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ <del>with no individual analyte RSD <math>&gt; 30\%</math></del>	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r > 0.995$ for each analyte		
				<del>non-linear - <math>COV \geq 0.990</math> (6 points shall be used for second order, 7 points shall be used for third order)</del>		
		Second-source calibration verification for PCB 1016/1260 mix	Once per five-point initial calibration	Mix within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for PCB 1016/1260 mix	Each initial calibration and calibration verifications	$\pm 3$ times standard deviation for each quantitation peak retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification for PCB 1016/1260 mix	Daily, before sample analysis	Results within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	Results within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.6-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8082	PCBs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.6-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS (1016/1260 mix)	One LCS per analytical batch	QC acceptance criteria, Table 7.2.6-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

**Table 7.2.6-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8082	PCBs	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.6-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for the surrogate apply J to all positive results  if the %R < LCL for the surrogate, apply J to all positive results, apply R to all non-detects  If the surrogate recovery is < 10%, apply R to all results
		MS/MSD (1016/1260 mix)	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.6-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.6-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



### **7.2.7 Method SW8141A-Organophosphorus Pesticides**

Method SW8141A is a GC method used to determine the concentrations of various organophosphorus pesticides. This analytical method involves extraction of the samples. An aliquot of the extract is injected into a GC and compounds in the GC effluent are detected with a flame photometric or nitrogen-phosphorus detector. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column. RLs for these pesticides are presented in Table 7.2.7-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.7-2 and 7.2.7-3.

**Table 7.2.7-1. RLs for Method SW8141A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Organophosphorus Pesticides SW8141A	Azinphos Methyl	1.0	µg/L	0.05	mg/kg
	Bolstar	0.7	µg/L	0.04	mg/kg
	Chlorpyrifos	0.7	µg/L	0.05	mg/kg
	Coumaphos	2.0	µg/L	0.10	mg/kg
	Demeton-o	1.2	µg/L	0.06	mg/kg
	Demeton-s	1.2	µg/L	0.06	mg/kg
	Diazinon	2.0	µg/L	0.10	mg/kg
	Dichlorovos	8.0	µg/L	0.04	mg/kg
	Disulfoton	0.7	µg/L	0.04	mg/kg
	Ethoprop	2.0	µg/L	0.10	mg/kg
	Fensulfothion	0.8	µg/L	0.04	mg/kg
	Fenthion	0.8	µg/L	0.05	mg/kg
	Merphos	2.0	µg/L	0.10	mg/kg
	Mevinphos	5.0	µg/L	0.25	mg/kg
	Naled	5.0	µg/L	0.25	mg/kg
	Parathion Methyl	1.2	µg/L	0.06	mg/kg
	Phorate	0.4	µg/L	0.02	mg/kg
	Ronnel	0.7	µg/L	0.04	mg/kg
	Stirophos	8.0	µg/L	0.40	mg/kg
	Tokuthion	0.7	µg/L	0.06	mg/kg
	Trichloronate	8.0	µg/L	0.40	mg/kg

**Table 7.2.7-2. QC Acceptance Criteria for Method SW8141A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8141A	Azinphos Methyl	50–150	≤ 30	40–160	≤ 50
	Bolstar	46–125	≤ 30	36–135	≤ 50
	Chlorpyrifos	75–125	≤ 30	65–135	≤ 50
	Coumaphos	71–147	≤ 30	61–157	≤ 50
	Demeton-o	50–150	≤ 30	40–160	≤ 50
	Demeton-s	50–150	≤ 30	40–160	≤ 50
	Diazinon	47–149	≤ 30	37–159	≤ 50
	Dichlorovos	49–125	≤ 30	39–135	≤ 50
	Disulfoton	50–150	≤ 30	40–160	≤ 50
	Ethoprop	75–125	≤ 30	65–135	≤ 50
	Fensulfothion	43–145	≤ 30	33–155	≤ 50
	Fenthion	25–125	≤ 30	25–135	≤ 50
	Merphos	75–144	≤ 30	65–154	≤ 50
	Mevinphos	33–125	≤ 30	25–135	≤ 50
	Naled	54–125	≤ 30	44–135	≤ 50
	Parathion Methyl	45–130	≤ 30	35–140	≤ 50
	Phorate	50–150	≤ 30	40–160	≤ 50
	Ronnel	75–125	≤ 30	65–135	≤ 50
	Stirophos	48–125	≤ 30	38–135	≤ 50
	Tokuthion	44–125	≤ 30	34–135	≤ 50
	Trichloronate	49–161	≤ 30	39–171	≤ 50
	<i>Surrogates:</i>				
	Tributyl Phosphate	67–136		57–146	
	Triphenyl Phosphate	65–134		55–144	

**Table 7.2.7-3. Summary of Calibration and QC Procedures for Method SW8141A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8141A	Organophosphorus pesticides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.7-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8141A	Organophosphorus pesticides	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.7-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.7-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

**Table 7.2.7-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8141A	Organophosphorus pesticides	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.7-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.7-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.7-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

## 7.2.8 Method SW8151A-Chlorinated Herbicides

Method SW8151A is a capillary GC method for determining selected chlorinated acid herbicides and related compounds. Samples are extracted then esterified. The esters are determined by GC employing an electron capture detector. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column. RLs for herbicides are presented in Table 7.2.8-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.8-2 and 7.2.8-3.

**Table 7.2.8-1. RLs for Method SW8151A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Chlorinated Phenoxy Acid Herbicides SW8151A	2,4-D	10	µg/L	0.2	mg/kg
	2,4-DB	20	µg/L	0.5	mg/kg
	2,4,5-T	20	µg/L	0.5	mg/kg
	2,4,5-TP	10	µg/L	0.2	mg/kg
	Dalapon	30	µg/L	0.8	mg/kg
	Dicamba	20	µg/L	0.5	mg/kg
	Dichlorprop	20	µg/L	0.5	mg/kg
	Dinoseb	3	µg/L	0.1	mg/kg
	MCPA	100	µg/L	10	mg/kg
	MCP	100	µg/L	15	mg/kg
	MCP	100	µg/L	15	mg/kg

**Table 7.2.8-2. QC Acceptance Criteria for Method SW8151A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8151A	2,4-D	39-120	≤ 30	32-131	≤ 50
	2,4-DB	44-120	≤ 30	42-145	≤ 50
	2,4,5-T	44-122	≤ 30	43-139	≤ 50
	2,4,5-TP	49-126	≤ 30	46-128	≤ 50
	Dalapon	40-120	≤ 30	22-125	≤ 50
	Dicamba	60-120	≤ 30	56-120	≤ 50
	Dichlorprop	68-122	≤ 30	72-142	≤ 50
	Dinoseb	28-115	≤ 30	20-131	≤ 50
	MCPA	62-144	≤ 30	65-120	≤ 50
	MCP	60-133	≤ 30	60-118	≤ 50
	<i>Surrogate:</i> 2,4-Dichlorophenylacetic acid	50-130		45-140	



**Table 7.2.8-3. Summary of Calibration and QC Procedures for Method SW8151A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8151A	Chlorinated Herbicides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r > 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	$\pm 3$ times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.8-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8151A	Chlorinated Herbicides	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.8-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.8-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.8-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results

**Table 7.2.8-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8151A	Chlorinated Herbicides	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.8-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first column result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.8-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

## 7.2.9 Method SW8260B-Volatile Organics

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260B. This method uses a capillary column GC/mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030B or SW5035) or other approved method (see table 7.1.1). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and RLs (using a 25 mL purge) for this method are listed in Table 7.2.9-1. Soil samples with higher contaminant levels can be extracted using methanol before purging. However, the RLs arising from the use of this preparatory method will be higher than those listed in Table 7.2.9-1 and the accuracy and precision requirements listed in Table 7.2.9-2 will not be met as well. Project specific DQOs and analytical protocols will need to be established if this preparatory method is used.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for BFB. The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50      15 percent to 40 percent of mass 95
- mass 75      30 percent to 60 percent of mass 95
- mass 95      base peak, 100 percent relative abundance
- mass 96      5 percent to 9 percent of mass 95
- mass 173     less than 2 percent of mass 174
- mass 174     greater than 50 percent of mass 95
- mass 175     5 percent to 9 percent of mass 174
- mass 176     greater than 95 percent, but less than 101 percent of mass 174
- mass 177     5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.9-2 and 7.2.9-3.

**Table 7.2.9-1. RLs for Method SW8260B**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B	1,1,1,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,1-TCA	1.0	µg/L	0.005	mg/kg
	1,1,2,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,2-TCA	1.0	µg/L	0.005	mg/kg
	1,1-DCA	1.0	µg/L	0.005	mg/kg
	1,1-DCE	1.0	µg/L	0.006	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichloropropane	1.0	µg/L	0.005	mg/kg
	1,2,4-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,4-Trimethylbenzene	1.0	µg/L	0.006	mg/kg
	1,2-DCA	0.5	µg/L	0.003	mg/kg
	1,2-DCB	1.0	µg/L	0.005	mg/kg
	1,2-Dibromo-3-chloropropane	2.0	µg/L	0.01	mg/kg
	1,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	1,2-Dibromoethane (EDB)	1.0	µg/L	0.005	mg/kg
	1,3,5-Trimethylbenzene	1.0	µg/L	0.005	mg/kg
	1,3-DCB	1.0	µg/L	0.006	mg/kg
	1,3-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,4-DCB	0.5	µg/L	0.002	mg/kg
	1-Chlorohexane	1.0	µg/L	0.005	mg/kg
	2,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	2-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	4-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	Acetone	10	µg/L	0.05	mg/kg
	Benzene	0.4	µg/L	0.002	mg/kg
	Bromobenzene	1.0	µg/L	0.005	mg/kg
	Bromochloromethane	1.0	µg/L	0.005	mg/kg
	Bromodichloromethane	0.5	µg/L	0.002	mg/kg
	Bromoform	1.0	µg/L	0.006	mg/kg
	Bromomethane	3.0	µg/L	0.01	mg/kg
	Carbon tetrachloride	1.0	µg/L	0.005	mg/kg
	Chlorobenzene	0.5	µg/L	0.002	mg/kg
	Chloroethane	1.0	µg/L	0.005	mg/kg
	Chloroform	0.3	µg/L	0.002	mg/kg
	Chloromethane	1.0	µg/L	0.005	mg/kg
	Cis-1,2-DCE	1.0	µg/L	0.005	mg/kg
	Cis-1,3-Dichloropropene	0.5	µg/L	0.003	mg/kg
	Dibromochloromethane	0.5	µg/L	0.003	mg/kg
	Dibromomethane	1.0	µg/L	0.005	mg/kg
	Dichlorodifluoromethane	1.0	µg/L	0.005	mg/kg
	Ethylbenzene	1.0	µg/L	0.005	mg/kg
	Hexachlorobutadiene	0.6	µg/L	0.003	mg/kg

**Table 7.2.9-1. Concluded**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B (concluded)	Isopropylbenzene	1.0	µg/L	0.005	mg/kg
	Methylene chloride	1.0	µg/L	0.005	mg/kg
	Methyl t-butyl ether (MTBE)	5.0	µg/L	0.02	mg/kg
	MEK (2-Butanone)	10	µg/L	0.02	mg/kg
	MIBK (methyl isobutyl ketone)	10	µg/L	0.02	mg/kg
	n-Butylbenzene	1.0	µg/L	0.005	mg/kg
	n-Propylbenzene	1.0	µg/L	0.005	mg/kg
	m,p-Xylene	2.0	µg/L	0.005	mg/kg
	Naphthalene	1.0	µg/L	0.005	mg/kg
	o-Xylene	1.0	µg/L	0.005	mg/kg
	p-Isopropyltoluene	1.0	µg/L	0.006	mg/kg
	Sec-Butylbenzene	1.0	µg/L	0.005	mg/kg
	Styrene	1.0	µg/L	0.005	mg/kg
	TCE	1.0	µg/L	0.005	mg/kg
	Tert-Butylbenzene	1.0	µg/L	0.005	mg/kg
	Tetrachloroethene	1.0	µg/L	0.005	mg/kg
	Toluene	1.0	µg/L	0.005	mg/kg
	Trans-1,2-DCE	1.0	µg/L	0.005	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Trichlorofluoromethane	1.0	µg/L	0.005	mg/kg
	Vinyl chloride	1.0	µg/L	0.005	mg/kg

**Table 7.2.9-2. QC Acceptance Criteria for Method SW8260B**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
SW8260B	1,1,1,2-Tetrachloroethane	81-129	≤ 20	74-125	≤ 30	2
	1,1,1-TCA	67-132	≤ 20	68-130	≤ 30	1
	1,1,2,2-Tetrachloroethane	63-128	≤ 20	59-140	≤ 30	3
	1,1,2-TCA	75-125	≤ 20	62-127	≤ 30	1
	1,1-DCA	69-133	≤ 20	73-125	≤ 30	1
	1,1-DCE	68-130	≤ 20	65-136	≤ 30	1
	1,1-Dichloropropene	73-132	≤ 20	70-135	≤ 30	1
	1,2,3-Trichlorobenzene	67-137	≤ 20	62-133	≤ 30	3
	1,2,3-Trichloropropane	73-124	≤ 20	63-130	≤ 30	3
	1,2,4-Trichlorobenzene	66-134	≤ 20	65-131	≤ 30	3
	1,2,4-Trimethylbenzene	74-132	≤ 20	65-135	≤ 30	3
	1,2-DCA	69-132	≤ 20	72-137	≤ 30	1
	1,2-DCB	71-122	≤ 20	74-120	≤ 30	3
	1,2-Dibromo-3-chloropropane	50-132	≤ 20	49-135	≤ 30	3
	1,2-Dichloropropane	75-125	≤ 20	71-120	≤ 30	1
	1,2-EDB	80-121	≤ 20	70-124	≤ 30	2
	1,3,5-Trimethylbenzene	74-131	≤ 20	65-133	≤ 30	3
	1,3-DCB	75-124	≤ 20	72-124	≤ 30	3
	1,3-Dichloropropane	73-126	≤ 20	76-123	≤ 30	2
	1,4-DCB	74-123	≤ 20	72-125	≤ 30	3
	1-Chlorohexane	70-125	≤ 20	60-135	≤ 30	2
	2,2-Dichloropropane	69-137	≤ 20	67-134	≤ 30	1
	2-Chlorotoluene	73-126	≤ 20	69-128	≤ 30	3
	4-Chlorotoluene	74-128	≤ 20	73-126	≤ 30	3
	Acetone	40-135	≤ 20	40-141	≤ 30	1
	Benzene	81-122	≤ 20	73-126	≤ 30	1
	Bromobenzene	76-124	≤ 20	66-121	≤ 30	3
	Bromochloromethane	65-129	≤ 20	71-127	≤ 30	1
	Bromodichloromethane	76-121	≤ 20	72-128	≤ 30	1
	Bromoform	69-128	≤ 20	66-137	≤ 30	2
	Bromomethane	53-141	≤ 20	45-141	≤ 30	1
	Carbon Tetrachloride	66-138	≤ 20	67-133	≤ 30	1
	Chlorobenzene	81-122	≤ 20	75-123	≤ 30	2
	Chloroethane	58-133	≤ 20	41-141	≤ 30	1
	Chloroform	69-128	≤ 20	72-124	≤ 30	1
	Chloromethane	56-131	≤ 20	51-129	≤ 30	1
	Cis-1,2-DCE	72-126	≤ 20	67-125	≤ 30	1
	Cis-1,3-Dichloropropene	69-131	≤ 20	72-126	≤ 30	1
	Dibromochloromethane	66-133	≤ 20	66-130	≤ 30	2
	Dibromomethane	76-125	≤ 20	73-128	≤ 30	1
	Dichlorodifluoromethane	53-153	≤ 20	34-136	≤ 30	1

Table 7.2.9-2. Concluded

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
SW8260B (Concluded)	Ethylbenzene	73-127	≤ 20	74-127	≤ 30	2
	Hexachlorobutadiene	67-131	≤ 20	53-142	≤ 30	3
	Isopropylbenzene	75-127	≤ 20	77-129	≤ 30	3
	m,p-Xylene	76-128	≤ 20	79-126	≤ 30	2
	Methylene chloride	63-137	≤ 20	63-137	≤ 30	1
	Methyl t-butyl ether (MtBE)	65-123	≤ 20	50-135	≤ 30	1
	MEK (2-Butanone)	49-136	≤ 20	40-135	≤ 30	1
	MIBK (methyl isobutyl ketone)	58-134	≤ 20	47-147	≤ 30	3
	n-Butylbenzene	69-137	≤ 20	65-138	≤ 30	3
	n-Propylbenzene	72-129	≤ 20	63-135	≤ 30	3
	Naphthalene	54-138	≤ 20	51-135	≤ 30	3
	o-Xylene	80-121	≤ 20	77-125	≤ 30	2
	p-Isopropyltoluene	73-130	≤ 20	75-133	≤ 30	3
	Sec-Butylbenzene	72-127	≤ 20	63-132	≤ 30	3
	Styrene	65-134	≤ 20	74-128	≤ 30	2
	TCE	70-127	≤ 20	77-124	≤ 30	1
	Tert-butylbenzene	70-129	≤ 20	65-132	≤ 30	3
	Tetrachloroethene	66-128	≤ 20	67-139	≤ 30	2
	Toluene	77-122	≤ 20	71-127	≤ 30	1
	Trans-1,2-DCE	63-137	≤ 20	66-134	≤ 30	1
	Trans-1,3-Dichloropropene	59-135	≤ 20	65-127	≤ 30	1
	Trichlorofluoromethane	57-129	≤ 20	49-139	≤ 30	1
	Vinyl Chloride	50-134	≤ 20	58-126	≤ 30	1
	<i>Surrogates:</i>					
	Dibromofluoromethane	85-115		65-135		
	Toluene-D8	81-120		84-116		
	4-Bromofluorobenzene	76-119		84-118		
	1,2-DCA-D4	72-119		52-149		
	<i>Internal Standards:</i>					
	Fluorobenzene					1
	Chlorobenzene-D5					2
	1,4-Dichlorobenzend-D					3



**Table 7.2.9-3. Summary of Calibration and QC Procedures for Method SW8260B**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.30^c$ and %RSD for RFs for CCCs $\leq 30\%$ and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes $\leq 15\%$ with no individual analyte RSD $> 30\%$		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression $r \geq 0.995$ for each analyte		
				option 3 non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm 0.06$ RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and after every 12 hours of analysis time	SPCCs average RF $\geq 0.30^c$ ; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within $\pm 20\%$ of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration

						verification
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**Table 7.2.9-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	Volatile Organics	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.9-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Internal Standards (ISs)	Each sample	Retention time $\pm 30$ seconds from retention time of the IS in the ICAL mid-point std. EICP area within -50% to +100% of area from IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply R to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M.
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.9-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.9-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL

**Table 7.2.9-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	Volatile Organics	Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 7.2.9)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.9-2	Correct problem then reextract and analyze sample	For the samples;  if the %R > UCL for a surrogate, apply J to all positive results  if the %R < LCL for a surrogate, apply J to all positive results; apply R to all non-detect results  If any surrogate recovery is <10%, apply R to all results
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.9-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

c. Except > 0.10 for bromoform, and > 0.10 for chloromethane and 1,1-dichloroethane

#### 7.2.10 Method SW8270C-Semivolatile Organics

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer. The RLs are listed in Table 7.2.10-1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 51      30 percent to 60 percent of mass 198
- mass 68      less than 2 percent of mass 69
- mass 70      less than 2 percent of mass 69
- mass 127     40 percent to 60 percent of mass 198
- mass 197     less than 1 percent of mass 198
- mass 198     base peak, 100 percent relative abundance
- mass 199     5 percent to 9 percent of mass 198
- mass 275     10 percent to 30 percent of mass 198
- mass 365     greater than 1 percent of mass 198
- mass 441     present, but less than mass 443
- mass 442     greater than 40 percent of mass 198
- mass 443     17 percent to 23 percent of mass 442

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.10-2 and 7.2.10-3.

**Table 7.2.10-1. RLs for Method SW8270C**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C	1,2,4-Trichlorobenzene	10.0	µg/L	0.7	mg/kg
	1,2-DCB	10.0	µg/L	0.7	mg/kg
	1,3-DCB	10.0	µg/L	0.7	mg/kg
	1,4-DCB	10.0	µg/L	0.7	mg/kg
	2,4-DNT	10.0	µg/L	0.7	mg/kg
	2,6-DNT	10.0	µg/L	0.7	mg/kg
	2-Chloronaphthalene	10.0	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10.0	µg/L	0.7	mg/kg
	2-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3,3'-Dichlorobenzidine	20.0	µg/L	1.3	mg/kg
	4-Bromophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Chloroaniline	20.0	µg/L	1.3	mg/kg
	4-Chlorophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Nitroaniline	50.0	µg/L	3.3	mg/kg
	Acenaphthylene	10.0	µg/L	0.7	mg/kg
	Acenaphthene	10.0	µg/L	0.7	mg/kg
	Anthracene	10.0	µg/L	0.7	mg/kg
	Benz (a) anthracene	10.0	µg/L	0.7	mg/kg
	Benzo (a) pyrene	10.0	µg/L	0.7	mg/kg
	Benzo (k) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10.0	µg/L	0.7	mg/kg
	Benzyl alcohol	20.0	µg/L	1.3	mg/kg
	Bis (2-chloroethoxy) methane	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroethyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroisopropyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-ethylhexyl) phthalate	10.0	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10.0	µg/L	0.7	mg/kg
	Chrysene	10.0	µg/L	0.7	mg/kg
	Di-n-butylphthalate	10.0	µg/L	0.7	mg/kg
	Di-n-octylphthalate	10.0	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10.0	µg/L	0.7	mg/kg
	Dibenzofuran	10.0	µg/L	0.7	mg/kg
	Diethyl phthalate	10.0	µg/L	0.7	mg/kg
	Dimethyl phthalate	10.0	µg/L	0.7	mg/kg
	Fluoranthene	10.0	µg/L	0.7	mg/kg
	Fluorene	10.0	µg/L	0.7	mg/kg
	Hexachlorobenzene	10.0	µg/L	0.7	mg/kg
	Hexachlorobutadiene	10.0	µg/L	0.7	mg/kg
	Hexachloroethane	10.0	µg/L	0.7	mg/kg
	Indeno (1,2,3-cd) pyrene	10.0	µg/L	0.7	mg/kg
	Isophorone	10.0	µg/L	0.7	mg/kg

**Table 7.2.10-1. Concluded**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C (concluded)	n-Nitrosodiphenylamine	10.0	µg/L	0.7	mg/kg
	n-Nitrosodi-n-propylamine	10.0	µg/L	0.7	mg/kg
	Naphthalene	10.0	µg/L	0.7	mg/kg
	Nitrobenzene	10.0	µg/L	0.7	mg/kg
	Phenanthrene	10.0	µg/L	0.7	mg/kg
	Pyrene	10.0	µg/L	0.7	mg/kg
Semivolatile organics Acid Extractables SW8270C	2,4,5-Trichlorophenol	50.0	µg/L	3.3	mg/kg
	2,4,6-Trichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dimethylphenol	10.0	µg/L	0.3	mg/kg
	2,4-Dinitrophenol	50.0	µg/L	3.3	mg/kg
	2-Chlorophenol	10.0	µg/L	0.3	mg/kg
	2-Methylphenol	10.0	µg/L	0.3	mg/kg
	2-Nitrophenol	10.0	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50.0	µg/L	3.3	mg/kg
	4-Chloro-3-methylphenol	20.0	µg/L	1.3	mg/kg
	4-Methylphenol	50.0	µg/L	2.0	mg/kg
	4-Nitrophenol	50.0	µg/L	1.6	mg/kg
	Benzoic acid	100	µg/L	5.0	mg/kg
	Pentachlorophenol	50.0	µg/L	3.3	mg/kg
	Phenol	10.0	µg/L	0.3	mg/kg

**Table 7.2.10-2. QC Acceptance Criteria for Method SW8270C**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C	1,2,4-Trichlorobenzene	37-120	≤ 20	44-125	≤ 30	2	4
	1,2-DCB	33-120	≤ 20	45-125	≤ 30	1	3
	1,3-DCB	32-120	≤ 20	39-125	≤ 30	1	3
	1,4-DCB	32-120	≤ 20	35-125	≤ 30	1	3
	2,4-DNT	51-120	≤ 20	48-125	≤ 30	3	4
	2,6-DNT	49-120	≤ 20	48-125	≤ 30	3	4
	2-Chloronaphthalene	49-120	≤ 20	45-125	≤ 30	3	4
	2-Methylnaphthalene	46-120	≤ 20	47-125	≤ 30	2	5
	2-Nitroaniline	48-120	≤ 20	44-125	≤ 30	3	2
	3,3'-Dichlorobenzidine	20-120	≤ 20	25-128	≤ 30	5	6
	3-Nitroaniline	20-126	≤ 20	27-125	≤ 30	3	2
	4-Bromophenyl phenyl ether	52-120	≤ 20	46-125	≤ 30	4	1
	4-Chloroaniline	20-120	≤ 20	25-125	≤ 30	2	5
	4-Chlorophenyl phenyl ether	50-120	≤ 20	47-125	≤ 30	3	4
	4-Nitroaniline	36-120	≤ 20	34-125	≤ 30	3	2
	Acenaphthylene	50-120	≤ 20	44-125	≤ 30	3	4
	Acenaphthene	47-120	≤ 20	46-125	≤ 30	3	4
	Anthracene	54-120	≤ 20	53-125	≤ 30	4	1
	Benz (a) anthracene	56-100	≤ 20	52-125	≤ 30	5	6
	Benzo (a) pyrene	53-120	≤ 20	50-125	≤ 30	6	6
	Benzo (b) fluoranthene	45-124	≤ 20	45-125	≤ 30	6	6
	Benzo (g,h,i) perylene	38-123	≤ 20	38-126	≤ 30	6	6
	Benzo (k) fluoranthene	45-124	≤ 20	45-125	≤ 30	6	6
	Benzyl alcohol	30-120	≤ 20	25-125	≤ 30	1	3
	Bis (2-chloroethoxy) methane	46-120	≤ 20	43-125	≤ 30	2	5
	Bis (2-chloroethyl) ether	37-120	≤ 20	38-125	≤ 30	1	3
	Bis (2-chloroisopropyl) ether	26-131	≤ 20	25-125	≤ 30	1	3
	Bis (2-ethylhexyl) phthalate	42-126	≤ 20	47-127	≤ 30	5	6
	Butyl benzyl phthalate	46-120	≤ 20	49-125	≤ 30	5	6
	Chrysene	55-120	≤ 20	53-125	≤ 30	5	6
	Di-n-butyl phthalate	54-120	≤ 20	56-125	≤ 30	4	1
	Di-n-octyl phthalate	37-137	≤ 20	41-132	≤ 30	5	6
	Dibenz (a,h) anthracene	42-127	≤ 20	41-125	≤ 30	6	6
	Dibenzofuran	54-120	≤ 20	51-125	≤ 30	3	4
	Diethyl phthalate	41-120	≤ 20	50-125	≤ 30	3	4
	Dimethyl phthalate	25-127	≤ 20	49-125	≤ 30	3	4
	Fluoranthene	54-120	≤ 20	54-125	≤ 30	4	1
	Fluorene	50-120	≤ 20	49-125	≤ 30	3	2



Table 7.2.10-2. Continued

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C (Continued)	Hexachlorobenzene	52-120	≤ 20	47-125	≤ 30	4	1
	Hexachlorobutadiene	27-120	≤ 20	40-125	≤ 30	2	5
	Hexachloroethane	28-120	≤ 20	34-125	≤ 30	1	3
	Indeno (1,2,3-c,d) pyrene	43-125	≤ 20	38-125	≤ 30	5	6
	Isophorone	50-120	≤ 20	43-125	≤ 30	2	5
	n-Nitrosodi-n-propylamine	34-128	≤ 20	40-125	≤ 30	1	3
	n-Nitrosodiphenylamine	48-120	≤ 20	49-125	≤ 30	4	1
	Naphthalene	39-120	≤ 20	40-125	≤ 30	2	5
	Nitrobenzene	44-120	≤ 20	41-125	≤ 30	2	4
	Phenanthrene	51-120	≤ 20	50-125	≤ 30	4	1
	Pyrene	49-128	≤ 20	46-125	≤ 30	5	6
	2,4,5-Trichlorophenol	49-120	≤ 20	49-125	≤ 30	3	1
	2,4,6-Trichlorophenol	49-126	≤ 20	43-125	≤ 30	3	1
	2,4-Dichlorophenol	48-120	≤ 20	45-125	≤ 30	2	5
	2,4-Dimethylphenol	28-120	≤ 20	32-125	≤ 30	2	5
	2,4-Dinitrophenol	25-130	≤ 20	25-132	≤ 30	3	4
	2-Chlorophenol	37-120	≤ 20	44-125	≤ 30	1	3
	2-Methylphenol	38-120	≤ 20	40-125	≤ 30	1	3
	2-Nitrophenol	39-123	≤ 20	42-125	≤ 30	2	4
	4,6-Dinitro-2-Methyl Phenol	40-130	≤ 20	29-137	≤ 30	4	1
	4-Chloro-3-Methyl Phenol	47-120	≤ 20	46-125	≤ 30	2	5
	4-Methylphenol	32-120	≤ 20	41-125	≤ 30	1	3
	4-Nitrophenol	20-120	≤ 20	25-138	≤ 30	3	2
	Benzoic Acid	20-120	≤ 20	25-125	≤ 30	2	5
	Pentachlorophenol	38-120	≤ 20	25-125	≤ 30	4	1
	Phenol	20-120	≤ 20	39-125	≤ 30	1	5

Table 7.2.10-2. Concluded

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Number
SW8270C (Concluded)	<i>Surrogates:</i>					
	2,4,6-Tribromophenol	42-124		36-126		1
	2-Fluorobiphenyl	48-120		43-125		2
	2-Fluorophenol	20-120		37-125		3
	Nitrobenzene-D5	41-120		37-125		4
	Phenol-D5	20-120		40-125		5
	Terphenyl-D14	51-135		32-125		6
	<i>Internal Standards:</i>					
	1,4-Dichlorobenzene-D4					1
	Naphthalene-D8					2
	Acenaphthene-D10					3
	Phenanthrene-D10					4
	Chrysene-D12					5
	Perylene-D12					6

**Table 7.2.10-3. Summary of Calibration and QC Procedures for Method SW8270C**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.050$ and %RSD for RFs for CCCs $\leq 30\%$ and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes $\leq 15\%$ with no individual analyte RSD $> 30\%$		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression $r \geq 0.995$ for each analyte		
				option 3 non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm 0.06$ RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF $\geq 0.050$ ; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within $\pm 20\%$ of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration



**Table 7.2.10-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8270C	Semi-Volatile Organics	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.10-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Internal Standards (ISs)	Each sample	Retention time $\pm 30$ seconds from retention time of the IS in the ICAL mid-point std. EICP area within -50% to +100% of area of IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply R to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M.
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.10-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.10-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL

								or (3) MS/MSD RPD > CL
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**Table 7.2.10-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8270C	Semi-Volatile Organics	Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 7.2.10)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.10-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results of analytes associated with the surrogate  if the %R < LCL for a surrogate, apply J to all positive results of analytes associated with the surrogate, apply R to all non-detect results of analytes associated with the surrogate  If any surrogate recovery is < 10%, apply R to all results of analytes associated with the surrogate
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.10-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.





#### **7.2.11 Method SW8290-Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans**

Method SW8290 is used to analyze for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in water, soil, and waste. This GC/MS method uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column GC/high resolution mass spectrometry techniques to separate and identify the analytes of interest. The sensitivity of the method is dependent on the level of matrix interference. Selected cleanup methods may be used to reduce or eliminate interferences. Target analytes may include all congener classes, tetra- through octa-dioxins and furans. Achieved detection limits vary according to matrix and analyte. Because of the extreme toxicity of these compounds, the analyst must take appropriate precautions during preparation and analysis to prevent accidental exposure. RLs are presented in Table 7.2.11-1.

The calibration, QC, corrective action, and data flagging requirements are given in Table 7.2.11-2.

**Table 7.2.11-1. RLs for Method SW8290**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Dioxins and Furans SW8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	0.01	ng/L	1.0	ng/kg
	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	0.01	ng/L	1.0	ng/kg
	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	0.05	ng/L	5.0	ng/kg
	2,3,7,8-Tetrachlorodibenzofuran (TCDF)	0.01	ng/L	1.0	ng/kg
	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	0.01	ng/L	1.0	ng/kg
	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	0.05	ng/L	1.0	ng/kg
	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	0.05	ng/L	5.0	ng/kg

**Table 7.2.11.2. Summary of Calibration and QC Procedures for Method SW8290**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8290	Dioxins/ Furans	Mass spectrometer tune	As per method SW8290, section 7.6.2	As per method SW8290, section 7.6.2	Retune instrument; verify	Apply R to the result for the specific analyte(s) for all samples associated with the tune
		Initial and continuing calibration	As per method SW8290, section 7.7	As per method SW8290, section 7.7	Correct problem then repeat calibration	Apply R to the result for the specific analyte(s) for all samples associated with the calibration
		Identification /retention times/ion ratios/signal to noise/interferences	As per method SW8290, section 7.8.4	As per method SW8290, section 7.8.4	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples associated with the condition
		System performance check	As per method SW8290, section 8.2	As per method SW8290, section 8.2	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the check
		Quality control checks	As per method SW8290, section 8.3	As per method SW8290, section 8.3	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the QC check
		Internal standard	As per method SW8290, section 8.4	As per method SW8290, section 8.4	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the internal standard
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.11-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



#### **7.2.12 Method SW8310–Polynuclear Aromatic Hydrocarbons**

Method SW8310 is used to determine the concentration of ppb levels of selected polynuclear aromatic hydrocarbons (PAHs) in groundwater and soils by HPLC. Samples are extracted then analyzed by direct injection. Detection is by ultraviolet and fluorescent detectors. RLs are listed in Table 7.2.12-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.12-2 and 7.2.12-3.

**Table 7.2.12-1. RLs for Method SW8310**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Polynuclear Aromatic Hydrocarbons SW8310	Acenaphthene	1.0	µg/L	0.2	mg/kg
	Acenaphthylene	1.0	µg/L	0.1	mg/kg
	Anthracene	1.0	µg/L	0.1	mg/kg
	Benzo (a) anthracene	0.1	µg/L	0.01	mg/kg
	Benzo (a) pyrene	0.2	µg/L	0.015	mg/kg
	Benzo (b) fluoranthene	0.2	µg/L	0.01	mg/kg
	Benzo (g,h,i) perylene	0.5	µg/L	0.05	mg/kg
	Benzo (k) fluoranthene	0.2	µg/L	0.01	mg/kg
	Chrysene	0.5	µg/L	0.1	mg/kg
	Dibenzo (a,h) anthracene	0.2	µg/L	0.01	mg/kg
	Fluoranthrene	1.0	µg/L	0.1	mg/kg
	Fluorene	2.0	µg/L	0.2	mg/kg
	Indeno (1,2,3-c,d) pyrene	0.2	µg/L	0.03	mg/kg
	Naphthalene	1.0	µg/L	0.2	mg/kg
	Phenanthrene	1.0	µg/L	0.1	mg/kg
	Pyrene	1.0	µg/L	0.1	mg/kg

**Table 7.2.12-2. QC Acceptance Criteria for Method SW8310**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8310	Acenaphthene	37-128	≤ 30	37-128	≤ 50
	Acenaphthylene	40-121	≤ 30	40-121	≤ 50
	Anthracene	41-120	≤ 30	47-125	≤ 50
	Benzo (a) Anthracene	49-120	≤ 30	50-120	≤ 50
	Benzo (a) Pyrene	45-120	≤ 30	40-133	≤ 50
	Benzo (b) Fluoranthene	51-120	≤ 30	57-121	≤ 50
	Benzo (g,h,i) Perylene	34-120	≤ 30	53-120	≤ 50
	Benzo (k) Fluoranthene	48-120	≤ 30	48-121	≤ 50
	Chrysene	50-120	≤ 30	55-120	≤ 50
	Dibenzo (a,h) Anthracene	33-120	≤ 30	47-120	≤ 50
	Fluoranthene	48-120	≤ 30	43-129	≤ 50
	Fluorene	42-128	≤ 30	46-120	≤ 50
	Indeno (1,2,3-c,d) Pyrene	47-120	≤ 30	56-134	≤ 50
	Naphthalene	33-120	≤ 30	48-120	≤ 50
	Phenathrene	40-120	≤ 30	57-126	≤ 50
	Pyrene	52-120	≤ 30	49-120	≤ 50
	<i>Surrogates:</i>				
	Terphenyl-D14	25-157		22-167	
	Decafluorobiphenyl	33-141		37-152	

**Table 7.2.12-3. Summary of Calibration and QC Procedures for Method SW8310**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8310	PAHs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD of average CF of all analytes ≤20% and average CF of individual analyte <30% or mean RSD for all analytes ≤20% with no individual analyte RSD > 30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification



**Table 7.2.12-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8310	PAHs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.12-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.12-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.12-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R



**Table 7.2.12-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8310	PAHs	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.12-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second Column Confirmation <sup>c</sup>	100% for all positive results	Same as for initial or primary analysis	Same as for initial or primary analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.12-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
- c. Use a second column or different detector

### **7.2.13 Method SW8321A-Chlorinated Herbicides by HPLC/MS**

Method SW8321A is a high performance liquid chromatography/mass spectrometry (HPLC/MS) method for determining selected chlorinated phenoxyacid herbicides and related compounds.

Samples are pH adjusted, extracted, and then separated by HPLC. The analysis can be performed for both the chlorinated phenoxyacid compounds (free acid form) and their esters without the use of hydrolysis or esterification in the extraction process. However, hydrolysis to the acid form is recommended because it simplifies the quantitation. A thermospray or other appropriate interface couples the LC to the MS for detection.

The method employs internal standards for quantitation. HPLC/MS utilizes an interface that substantially reduces the solvent introduced into the mass spectrometer. The small amount of solvent or mobile phase that is present after sample introduction into the mass spectrometer acts as a quasi chemical ionization reagent and the resulting spectra are chemical-ionization like in both the positive and negative ionization modes. Fragmentation patterns seen in traditional electron impact spectrometers do not occur. Tuning may be accomplished using polyethylene glycol (PEG) 400 or other tuning standards as recommended by the manufacturer's specifications or other documented source.

RLs for herbicides are presented in Table 7.2.13-1. The calibration, QC, corrective action, and data flagging requirements are given in tables 7.2.13-2 and 7.2.13-3.

**Table 7.2.13-1. RLs for Method SW8321A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Chlorinated Phenoxyacid Herbicides SW8321A	2,4-D	5.0	µg/L	0.2	mg/kg
	2,4-DB	5.0	µg/L	0.2	mg/kg
	2,4,5-T	5.0	µg/L	0.2	mg/kg
	2,4,5-TP	5.0	µg/L	0.2	mg/kg
	Dalapon	20.0	µg/L	0.4	mg/kg
	Dicamba	5.0	µg/L	0.3	mg/kg
	Dichlorprop	5.0	µg/L	0.2	mg/kg
	Dinoseb	3.0	µg/L	0.1	mg/kg
	MCPA	5.0	µg/L	0.2	mg/kg
	MCP	5.0	µg/L	0.2	mg/kg

**Table 7.2.13-2. QC Acceptance Criteria for Method SW8321A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8321A	2,4-D	50-115	≤ 30	55-130	≤ 50
	2,4-DB	55-120	≤ 30	60-135	≤ 50
	2,4,5-T	55-130	≤ 30	55-130	≤ 50
	2,4,5-TP	60-130	≤ 30	55-135	≤ 50
	Dalapon	50-125	≤ 30	50-125	≤ 50
	Dicamba	45-115	≤ 30	40-135	≤ 50
	Dichlorprop	50-120	≤ 30	60-130	≤ 50
	Dinoseb	50-125	≤ 30	45-135	≤ 50
	MCPA	55-120	≤ 30	55-135	≤ 50
	MCP	60-125	≤ 30	60-135	≤ 50
	<i>Surrogates<sup>a</sup>:</i>				
	4-Nitrophenol	25-125	≤ 30	25-140	≤ 50
	Dichloroacetic acid (DCAA)		≤ 30		≤ 50
	<i>Internal Standards<sup>a</sup>:</i>				
	2,6-Dinitrotoluene-D3				
	Atrazine-D5				
	Acifluorfen				

a. The choices for surrogates and internal standards will depend on the ionization mode employed. Other compounds may be used. However, the laboratory must provide documentation supporting their choices and surrogate recovery limits.

**Table 7.2.13-3. Summary of Calibration and QC Procedures for Method SW8321A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8321A	Chlorinated Herbicides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear- mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$ linear least squares regression $r > 0.985$ for each analyte Non-linear regression - COD $> 0.97$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm 0.06$ RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	All analytes within $\pm 30\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.13-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst

**Table 7.2.13-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8321A	Chlorinated Herbicides	Internal Standard (IS)	Each sample	Retention time $\pm 30$ seconds from average retention time in the ICAL.  IS within $\pm 30\%$ of average value in the ICAL.	Inspect mass spectrometer and HPLC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply R to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M.
		Method Blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.13-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.13-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL

**Table 7.2.13-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8321A	Chlorinated Herbicides	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.13-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results of analytes associated with the surrogate  If the %R < LCL for a surrogate, apply J to all positive results of analytes associated with the surrogate, apply R to all non-detect results of analytes associated with the surrogate  If any surrogate recovery is < 10%, apply R to all results of analytes associated with the surrogate
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.13-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and low standard	None	none	none	Apply F to all results between MDL and low standard

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



#### **7.2.14 Method SW8330—Explosive Residues**

Method SW8330 provides HPLC conditions for the detection of ppb levels of certain explosive residues in a water, soil, and sediment matrix. Prior to using this method, appropriate sample preparation techniques must be used.

Two low-level preparatory methods exist, salting out and solid phase extraction (SPE). In the salting-out method, aqueous samples of low concentration are extracted by a salting-out extraction procedure with no evaporation. SPE (Method SW3535A) is a procedure for isolating organic compounds from aqueous samples using solid-phase extraction media. SPE is the preferred method due to the increased accuracy and precision it gives over salting out. An aliquot of the extract is separated on a C-18 reverse-phase column, determined at 254 nm, and confirmed on a cyanide reverse-phase column.

In the high-level direct injection method, aqueous samples of higher concentration can be diluted, filtered, separated on a C-18 reverse-phase column, determined at 254 nm, and confirmed on a cyanide reverse-phase column.

Soil and sediment samples are extracted in an ultrasonic bath and filtered before chromatography.

RLs are listed in Table 7.2.14-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.14-2 and 7.2.14-3.

**Table 7.2.14-1. RLs for Method SW8330**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Explosive Residues SW8330	1,3,5- TNB	1.0	µg/L	0.25	mg/kg
	1,3- DNB	1.0	µg/L	0.25	mg/kg
	2,4,6- TNT	1.0	µg/L	0.25	mg/kg
	2,4-DNT	1.0	µg/L	0.25	mg/kg
	2,6-DNT	1.0	µg/L	0.26	mg/kg
	HMX	1.0	µg/L	2.2	mg/kg
	m-Nitrotoluene	1.0	µg/L	0.25	mg/kg
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	1.0	µg/L	0.65	mg/kg
	Nitrobenzene	1.0	µg/L	0.26	mg/kg
	o-Nitrotoluene	1.0	µg/L	0.25	mg/kg
	p-Nitrotoluene	1.0	µg/L	0.25	mg/kg
	RDX	1.0	µg/L	1.0	mg/kg

**Table 7.2.14-2. QC Acceptance Criteria for Method SW8330**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8330	1,3,5-TNB	64-139	≤ 30	54-136	≤ 50
	1,3-DNB	47-158	≤ 30	79-124	≤ 50
	2,4,6-TNT	52-143	≤ 30	55-142	≤ 50
	2,4-DNT	61-135	≤ 30	56-141	≤ 50
	2,6-DNT	60-137	≤ 30	77-122	≤ 50
	HMX	51-161	≤ 30	72-134	≤ 50
	m-Nitrotoluene	48-132	≤ 30	52-133	≤ 50
	Methyl-2,4,6-Trinitrophenylnitramine (Tetryl)	22-174	≤ 30	25-142	≤ 50
	Nitrobenzene	49-138	≤ 30	49-154	≤ 50
	o-Nitrotoluene	43-133	≤ 30	59-136	≤ 50
	p-Nitrotoluene	48-132	≤ 30	77-124	≤ 50
	RDX	81-120	≤ 30	74-126	≤ 50
	<i>Surrogates<sup>a</sup>:</i>				

- a. Use an analyte and its LCS limit from the method that is not expected to be present in the sample as the surrogate.

**Table 7.2.14-3. Summary of Calibration and QC Procedures for Method SW8330**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8330	Explosives	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD of average CF of all analytes ≤20% and average CF of individual analyte <30% or mean RSD for all analytes ≤20% with no individual analyte RSD > 30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear - least squares regression $r \geq 0.995$ for each analyte		
				non-linear - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
			After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.14-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8330	Explosives	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.14-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.14-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.14-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results  if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R

						to all results
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**Table 7.2.14-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8330	Explosives	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.14-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Second Column Confirmation	100% for all positive results	Same as for initial or primary analysis	Same as for initial or primary analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed.  Apply J if RPD >40% from first result
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.14-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.15 Method SW6010B-Trace Elements (Metals) by Inductively Coupled Plasma Atomic Emission Spectroscopy for Water and Soil**

Samples are analyzed for trace elements or metals using method SW6010B for water and soils. Analysis for most metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICPES). The elements and corresponding RLs for this method are listed in Table 7.2.15-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.15-2 and 7.2.15-3.

**Table 7.2.15-1. RLs for Method SW6010B**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg



**Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤ 20	79-120	≤ 30
	Antimony	80-120	≤ 20	80-120	≤ 30
	Arsenic	80-120	≤ 20	80-120	≤ 30
	Barium	80-120	≤ 20	80-120	≤ 30
	Beryllium	80-120	≤ 20	80-120	≤ 30
	Cadmium	80-120	≤ 20	80-120	≤ 30
	Calcium	80-120	≤ 20	80-120	≤ 30
	Chromium	80-120	≤ 20	80-120	≤ 30
	Cobalt	80-120	≤ 20	80-120	≤ 30
	Copper	80-120	≤ 20	80-120	≤ 30
	Iron	80-120	≤ 20	80-120	≤ 30
	Lead	80-120	≤ 20	80-120	≤ 30
	Magnesium	80-120	≤ 20	80-120	≤ 30
	Manganese	80-120	≤ 20	80-120	≤ 30
	Molybdenum	79-120	≤ 20	80-120	≤ 30
	Nickel	80-120	≤ 20	80-120	≤ 30
	Potassium	80-120	≤ 20	80-120	≤ 30
	Selenium	80-120	≤ 20	80-120	≤ 30
	Silver	80-120	≤ 20	75-120	≤ 30
	Sodium	80-120	≤ 20	80-120	≤ 30
	Thallium	80-120	≤ 20	80-120	≤ 30
	Vanadium	80-120	≤ 20	80-120	≤ 30
	Zinc	80-120	≤ 20	80-120	≤ 30

**Table 7.2.15-3. Summary of Calibration and QC Procedures for Method SW6010B**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6010B	ICP Metals	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be $\geq 0.995$	If applicable, correct problem and repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Initial calibration verification (second source)	Daily after initial calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within $\pm 10\%$ of expected value and RSD of replicate integrations $< 5\%$	Repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Calibration blank	After every calibration verification	No analytes detected $\geq$ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.15-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with $\pm 50\%$ of expected value	Correct problem then reanalyze	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Linear range calibration (high) check standard	Every three months	Analyte within $\pm 10\%$ of expected value	Correct problem then reanalyze or re-set linear range	Apply J to specific analyte(s) for all results not within linear range

**Table 7.2.15-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6010B	ICP Metals	Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		Interference check solution (ICS)	At the beginning of an analytical run	Within $\pm 20\%$ of expected value	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.15-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test	Each new sample matrix, at least once per analytical batch (only applicable for analytes with concentrations $\geq 50X$ MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform post digestion spike addition	Apply J to all sample results for specific analyte from the same matrix in the batch if either of following exist: (1) dilution test not run and batch had analyte concentrations $\geq 50X$ MDL (2) %D $\geq 10$ and post digestion spike not performed

**Table 7.2.15-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6010B	ICP Metals	Post digestion spike addition	When dilution test fails or if an analyte's concentration for all samples in a batch is less than 50X MDL	Recovery within 75-125% of expected results	Check for instrumental problem then reanalyze post digestion spike addition if appropriate	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition  If post digestion spike addition recovery is < 10%, apply R to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.15-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.15-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.16 Method SW6020-Trace Elements (Metals) by Inductively Coupled Plasma Mass Spectrometry for Water and Soil**

Samples are analyzed for trace elements or metals using method SW6020 for water and soils. Analysis for total (i.e., acid leachable) metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The elements and RLs for this method are listed in Table 7.2.16-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.16-2 and 7.2.16-3.

**Table 7.2.16-1. RLs for Method SW6020**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
ICP Screen for Metals SW6020	Aluminum	0.02	mg/L	2.0	mg/kg
	Antimony	0.001	mg/L	0.10	mg/kg
	Arsenic	0.02	mg/L	2.0	mg/kg
	Barium	0.003	mg/L	0.30	mg/kg
	Beryllium	0.003	mg/L	0.30	mg/kg
	Cadmium	0.002	mg/L	0.20	mg/kg
	Chromium	0.004	mg/L	0.40	mg/kg
	Cobalt	0.008	mg/L	0.80	mg/kg
	Copper	0.006	mg/L	0.60	mg/kg
	Lead	0.002	mg/L	0.20	mg/kg
	Manganese	0.002	mg/L	0.20	mg/kg
	Nickel	0.002	mg/L	0.20	mg/kg
	Silver	0.002	mg/L	0.20	mg/kg
	Thallium	0.0002	mg/L	0.02	mg/kg
	Zinc	0.025	mg/L	2.5	mg/kg

**Table 7.2.16-2. QC Acceptance Criteria for Method SW6020**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6020	Aluminum	80–120	≤ 15	80–120	≤ 25
	Antimony	80–120	≤ 15	80–120	≤ 25
	Arsenic	80–120	≤ 15	80–120	≤ 25
	Barium	80–120	≤ 15	80–120	≤ 25
	Beryllium	80–120	≤ 15	80–120	≤ 25
	Cadmium	80–120	≤ 15	80–120	≤ 25
	Chromium	80–120	≤ 15	80–120	≤ 25
	Cobalt	80–120	≤ 15	80–120	≤ 25
	Copper	80–120	≤ 15	80–120	≤ 25
	Lead	80–120	≤ 15	80–120	≤ 25
	Manganese	80–120	≤ 15	80–120	≤ 25
	Nickel	80–120	≤ 15	80–120	≤ 25
	Silver	80–120	≤ 15	80–120	≤ 25
	Thallium	80–120	≤ 15	80–120	≤ 25
	Zinc	80–120	≤ 15	80–120	≤ 25

**Table 7.2.16-3. Summary of Calibration and QC Procedures for Method SW6020**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6020	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	SW6020 paragraph 5.8	Retune instrument then reanalyze tuning solution	Apply R to all results for all analytes for all samples associated with the MS tuning
		Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be $\geq 0.995$	If applicable, correct problem and repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected $\geq$ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) above the RL in all samples associated with the blank
		Initial Calibration verification (Second source standard)	After initial calibration before beginning a sample run - at a concentration other than used for calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for the specific analyte(s) in all samples
		Continuing Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with $\pm 50\%$ of expected value	Correct problem then reanalyze	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Linear range calibration (high) check standard	Every three months	Analyte within $\pm 10\%$ of expected value	Correct problem then reanalyze or re-set linear range	Apply J to specific analyte(s) for all results not within linear range



**Table 7.2.16-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6020	ICP/MS Metals	Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or once during an 12 hour period, whichever is more frequent	ICS-A All non-spiked analytes < RL unless they are a verified trace impurity from one of the spiked analytes  ICS-AB Within $\pm 20\%$ of true value	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.16-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test	Each matrix in a analytical batch (only applicable for analytes with concentrations $\geq 100X$ MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform post digestion spike addition	Apply J to all sample results where analyte concentrations were $> 100X$ MDL and either dilution test not performed or %D > 10 but post digestion spike test was not performed
		Post digestion spike addition	When dilution test fails Or if an analyte's concentration for all samples in a batch is less than $100X$ MDL	Recovery within 75-125% of expected results	Dilute the sample; reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the failed post digestion spike addition

**Table 7.2.16-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW6020	ICP/MS Metals	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.16-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Internal Standards (ISs)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in method SW6020, section 8.3	Apply R to all results for specific analyte(s) in all samples associated with the IS.
		IDL study	Every three months	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.16-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		MDL study	Every 12 months			
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.16-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.17 Method SW7041–Graphite Furnace Atomic Absorption (Antimony)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted then discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the antimony. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.17-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.17-2 and 7.2.17-3.

**Table 7.2.17-1. RLs for Method SW7041**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7041	Antimony	0.005	mg/L	0.5	mg/kg

**Table 7.2.17-2. QC Acceptance Criteria for Method SW7041**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7041	Antimony	75–125	≤ 15	75–125	≤ 30

**Table 7.2.17-3. Summary of Calibration and QC Procedures for Method SW7041**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7041	Antimony	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq RL$	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.17-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq RL$	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.17-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7041	Antimony	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.17-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.17-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7041	Antimony	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.17-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.17-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.18 Method SW7060A–Graphite Furnace Atomic Absorption (Arsenic)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the arsenic. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.18-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.18-2 and 7.2.18-3.



**Table 7.2.18-1. RLs for Method SW7060A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7060A	Arsenic	0.005	mg/L	0.5	mg/kg

**Table 7.2.18-2. QC Acceptance Criteria for Method SW7060A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7060A	Arsenic	74-120	≤ 15	74-120	≤ 30

**Table 7.2.18-3. Summary of Calibration and QC Procedures for Method SW7060A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7060A	Arsenic	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.18-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.18-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7060A	Arsenic	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.18-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.18-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7060A	Arsenic	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.18-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.18-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.19 Method SW7131A–Graphite Furnace Atomic Absorption (Cadmium)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Cadmium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analyzes are listed in Table 7.2.19-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.19-2 and 7.2.19-3.

**Table 7.2.19-1. RLs for Method SW7131A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7131A	Cadmium	0.001	mg/L	0.1	mg/kg

**Table 7.2.19-2. QC Acceptance Criteria for Method SW7131A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7131A	Cadmium	80-122	≤ 15	80-122	≤ 30

**Table 7.2.19-3. Summary of Calibration and QC Procedures for Method SW7131A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7131A	Cadmium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq RL$	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.19-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq RL$	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.19-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7131A	Cadmium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.19-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations $\geq 25X$ MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations $> 25X$ MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than $25X$ MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range



**Table 7.2.19-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7131A	Cadmium	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.19-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.19-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.20 Method SW7191–Graphite Furnace Atomic Absorption (Chromium)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Chromium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analyzes are listed in Table 7.2.20-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.20-2 and 7.2.20-3.

**Table 7.2.20-1. RLs for Method SW7191**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7191	Chromium	0.005	mg/L	0.5	mg/kg

**Table 7.2.20-2. QC Acceptance Criteria for Method SW7191**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7191	Chromium	80-121	≤ 15	80-121	≤ 30

**Table 7.2.20-3. Summary of Calibration and QC Procedures for Method SW7191**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7191	Chromium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.20-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.20-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7191	Chromium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.20-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.20-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7191	Chromium	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.20-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.20-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

## 7.2.21 Method SW7196A-Hexavalent Chromium (Colorimetric)

Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically. RLs for this method are listed in Table 7.2.21-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.21-2 and 7.2.21-3.

**Table 7.2.21-1. RLs for Method SW7196A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7196A	Hexavalent Chromium	0.5	mg/L	1.0	mg/kg

**Table 7.2.21-2. QC Acceptance Criteria for Method SW7196A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7196A	Hexavalent Chromium	86-117	≤ 15	86-117	≤ 30

**Table 7.2.21-3. Summary of Calibration and QC Procedures for Method SW7196A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7196A	Hexavalent Chromium	Multipoint calibration curve (minimum three standards and a blank)	Initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Second-source calibration verification	After each new stock standard preparation	Analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Calibration verification	After every 15 samples and at the end of the analysis sequence	Chromium within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration and reanalyze all samples since last successful calibration	Apply R to the specific analyte result in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.21-2	Recalculate results; locate and fix problem with system and then rerun demonstration	Apply R to the specific analyte result for all samples analyzed by the analyst
		Verification check to ensure lack of reducing condition and/or interference	Once for every sample matrix analyzed	Spike recovery between 85-115%	If check indicates interference, dilute and reanalyze sample persistent interference indicates the need to use and alternate method	Apply R to the specific analyte result for all samples analyzed since the last acceptable verification check
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.21-1	none	Apply R to all specific analyte results for all samples analyzed



**Table 7.2.21-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7196A	Chromium	Method blank	One per analytical batch	No analyte detected > RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the specific analyte result for all samples in the associated analytical batch
		LCS	One LCS per analytical batch	QC acceptance criteria, Table 7.2.21-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.21-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if;(1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.22 Method SW7421–Graphite Furnace Atomic Absorption (Lead)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Lead. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.22-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.22-2 and 7.2.22-3.

**Table 7.2.22-1. RLs for Method SW7421**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7421	Lead	0.005	mg/L	0.5	mg/kg

**Table 7.2.22-2. QC Acceptance Criteria for Method SW7421**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7421	Lead	74-124	≤ 15	74-124	≤ 30

**Table 7.2.22-3. Summary of Calibration and QC Procedures for Method SW7421**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7421	Lead	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.22-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.22-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7421	Lead	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.22-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.22-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7421	Lead	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.22-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.22-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### **7.2.23 Method SW7470A/SW7471A–Mercury Manual Cold-Vapor Technique**

Water and soil samples are analyzed for mercury using methods SW7470A and SW7471A, respectively. This method is a cold-vapor, flameless atomic absorption (AA) technique based on the absorption of radiation by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Mercury concentration is measured as a function of absorbance. The RLs for these methods are listed in Table 7.2.23-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.23-2 and 7.2.23-3.

**Table 7.2.23-1. RLs for Method SW7470A/SW7471A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7470A (W) SW7471A (S)	Mercury	0.001	mg/L	0.1	mg/kg

**Table 7.2.23-2. QC Acceptance Criteria for Method SW7470A/SW7471A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7470A/SW7471A	Mercury	85-115	≤ 15	83-118	≤ 30



**Table 7.2.23-3. Summary of Calibration and QC Procedures for Method  
SW7470A/SW7471A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7470A SW7471A	Mercury	Initial multipoint calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.23-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.23-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7470A SW7471A	Mercury	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.23-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations $\geq 25X$ MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	None	Apply J to all sample results if batch had sample(s) with concentrations $> 25X$ MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.23-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if: (1) %R for MS or MSD > UCL or (2) %R for MS or MSD < LCL or (3) MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.23-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



#### **7.2.24 Method SW7521–Graphite Furnace Atomic Absorption (Nickel)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the nickel. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.24-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.24-2 and 7.2.24-3.

**Table 7.2.24-1. RLs for Method SW7521**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7521	Nickel	0.005	mg/L	0.05	mg/kg

**Table 7.2.24-2. QC Acceptance Criteria for Method SW7521**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7521	Nickel	75-125	≤ 15	75-125	≤ 30

**Table 7.2.24-3. Summary of Calibration and QC Procedures for Method SW7521**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7521	Nickel	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.24-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.24-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7521	Nickel	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.24-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.24-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7521	Nickel	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.24-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.24-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



#### **7.2.25 Method SW7740-Graphite Furnace Atomic Absorption (Selenium)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are prepared as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Selenium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.25-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.25-2 and 7.2.25-3.

**Table 7.2.25-1. RLs for Method SW7740**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7740	Selenium	0.005	mg/L	0.5	mg/kg

**Table 7.2.25-2. QC Acceptance Criteria for Method SW7740**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7740	Selenium	73-122	≤ 15	73-122	≤ 30

**Table 7.2.25-3. Summary of Calibration and QC Procedures for Method SW7740**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7740	Selenium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.25-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.25-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7740	Selenium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.25-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.25-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7740	Selenium	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.25-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.25-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.26 Method SW7841–Graphite Furnace Atomic Absorption (Thallium)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Thallium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.26-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.26-2 and 7.2.26-3.

**Table 7.2.26-1. RLs for Method SW7841**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7841	Thallium	0.002	mg/L	0.1	mg/kg

**Table 7.2.26-2. QC Acceptance Criteria for Method SW7841**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7841	Thallium	78-123	≤ 15	78-123	≤ 30

**Table 7.2.26-3. Summary of Calibration and QC Procedures for Method SW7841**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7841	Thallium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.26-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch



**Table 7.2.26-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7841	Thallium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.26-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.26-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7841	Thallium	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.26-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.26-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.27 Method SW7911-Graphite Furnace Atomic Absorption (Vanadium)**

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Vanadium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 7.2.27-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.27-2 and 7.2.27-3.

**Table 7.2.27-1. RLs for Method SW7911**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7911	Vanadium	0.004	mg/L	0.4	mg/kg

**Table 7.2.27-2. QC Acceptance Criteria for Method SW7911**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7911	Vanadium	78-123	≤ 15	78-123	≤ 30

**Table 7.2.27-3. Summary of Calibration and QC Procedures for Method SW7911**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7911	Vanadium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected $\geq$ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.27-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

**Table 7.2.27-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7911	Vanadium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.27-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations >25X MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform recovery test	Apply J to all sample results if batch had sample(s) with concentrations >25X MDL and either of following exist: (1) dilution test not run (2) %D $\geq 10$ and recovery test not performed
		Recovery test	When dilution test fails Or if analyte concentration for all samples in a batch is less than 25X MDL	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

**Table 7.2.27-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7911	Vanadium	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.27-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.27-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

#### **7.2.28 Method SW9010B/SW9012A-Total Cyanide and Cyanide Amenable to Chlorination**

Water and waste samples are analyzed for total cyanide using method SW9010B or SW9012A. These methods are equivalent in principle of analysis; SW9010B is a manual procedure, and SW9012A is an automated procedure.

Both methods are used to determine the concentration of inorganic cyanide in aqueous wastes and leachates. The methods detect inorganic cyanides that are present as either sample soluble salts or complex radicals. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The cyanide is released by refluxing the sample with a strong acid and catalyst and distillation. Total cyanide in soils is determined after acidification of the soil and distillation. The cyanide ion in the absorbing solution is then determined by spectrophotometry for method SW9010B and by automated colorimetry for method SW9012A. RLs for cyanide are listed in Table 7.2.28-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.28-2 and 7.2.28-3.



**Table 7.2.28-1. RLs for Method SW9010B/SW9012A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW9010B/SW9012A	Total cyanide	0.02	mg/L	0.5	mg/kg

**Table 7.2.28-2. QC Acceptance Criteria for Method SW9010B/SW9012A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (%R)	Precision Soil (% RPD)
SW9010B SW9012A	Total cyanide	79-114	≤ 20	75-125	≤ 30

**Table 7.2.28-3. Summary of Calibration and QC Procedures for Method  
SW9010B/SW9012A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW9010B/ SW9012A	Cyanide	Multipoint calibration curve (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Distilled standards (one high and one low)	Once per multipoint calibration	Cyanide within $\pm 10\%$ of true value	Correct problem then repeat distilled standards	Apply R to all results for the specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per stock standard preparation	Cyanide within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply R to the result for the specific analyte for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.28-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to the specific analyte result for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the result for the specific analyte in all samples in the associated analytical batch

**Table 7.2.28-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW9010B/ SW9012A	Cyanide	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.22-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For the specific analyte in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.22-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if: (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.22-1	none	Apply R to all results for the specific analyte in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### **7.2.29 Method SW9056A–Common Anions**

This method addresses the sequential determination of the anions chloride, fluoride, bromide, nitrate, nitrite, phosphate, and sulfate in aqueous samples, aqueous extracts of solids, and the collection solutions from the bomb combustion of solid waste samples.

A small volume of aqueous sample is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of eluent. For aqueous extracts of solid samples, AFCEE recommends using the procedure listed in section 11.7 of EPA Method 300.0 (a 10-fold dilution of the solid sample with reagent grade water).

The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn (guard) column and a separator column, are packed with a low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

RLs are listed in Table 7.2.29-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.29-2 and 7.2.29-3.

**Table 7.2.29-1. RLs for Method SW9056A**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Common Anions SW9056A	Bromide	0.5	mg/L	5	mg/kg
	Chloride	1.0	mg/L	10	mg/kg
	Fluoride	1.0	mg/L	10	mg/kg
	Nitrate	1.0	mg/L	10	mg/kg
	Nitrite	1.0	mg/L	10	mg/kg
	Phosphate	1.0	mg/L	10	mg/kg
	Sulfate	1.0	mg/L	10	mg/kg

**Table 7.2.29-2. QC Acceptance Criteria for Method SW9056A**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW9056A	Bromide	85-115	≤ 20	70-130	≤ 30
	Chloride	85-115	≤ 20	70-130	≤ 30
	Fluoride	85-115	≤ 20	70-130	≤ 30
	Nitrate	85-115	≤ 20	70-130	≤ 30
	Nitrite	85-115	≤ 20	70-130	≤ 30
	Phosphate	85-115	≤ 20	70-130	≤ 30
	Sulfate	85-115	≤ 20	70-130	≤ 30

**Table 7.2.29-3. Summary of Calibration and QC Procedures for Method SW9056A**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW9056	Common anions	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient $\geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	$\pm 3$ times standard deviation for each analyte retention time over 8 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis or when eluent is changed	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

**Table 7.2.29-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW9056	Common anions	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.29-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.29-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Duplicate	One per every 10 samples	%D $\leq$ 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results

**Table 7.2.29-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW9056	Common anions	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.29-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.29-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



### **7.2.30 Method 314.0–Perchlorate Anion**

This method addresses the determination of the perchlorate anion in water samples as well as in aqueous extracts of soil samples using ion chromatography.

A large (approximately 1.0 mL) volume of sample is introduced into an ion chromatograph. Perchlorate is separated and measured using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

The method requires the use of a conductivity detector to monitor sample matrix conductivity and to determine if sample pretreatment is required. Pretreatment must be performed whenever the conductivity exceeds the laboratory determined Matrix Conductivity Threshold (MCT) and can consist of dilution and/or use of specific pretreatment cartridges or columns designed to remove matrix interferences. The MCT is the matrix conductance where the calculated A/H ratio percent difference ( $PD_{A/H}$ ) for the perchlorate peak exceeds 20 %.

An analytical batch is a sequence of samples which are analyzed within a 30 hour period and include no more than 20 field samples. An analytical batch must also include all required QC samples, which do not contribute to the maximum field sample total of 20. The required QC samples include:

- Instrument Performance Check Standard (IPC)
- Laboratory Reagent Blank (LRB)
- Initial Calibration Check Standard (ICCS) (also known as ICV)
- Laboratory Fortified Blank (LFB) (also known as LCS)
- Continuing Calibration Check Standard (CCCS) (also known as CCV)
- End Calibration Check Standard (ECCS) (also known as CCV)
- Laboratory Fortified Sample Matrix (LFM) (also known as MS)
- Duplicate of the LFM (also known as MSD). If no MS/MSD has been designated, a field duplicate or laboratory duplicate may be used.
- If pretreated samples are included in batch, Pretreated LRB, Pretreated LFB, Pretreated LFM (for each pretreated matrix)

RLs are listed in Table 7.2.30-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.30-2 and 7.2.30-3.

**Table 7.2.30-1. RLs for Method 314.0**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
314.0	Perchlorate	4.0	µg/L	50	µg/kg

**Table 7.2.30-2. QC Acceptance Criteria for Method 314.0**

Method	Sample	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
314.0	Perchlorate	80 -120	≤ 15	70 - 130	≤ 30

**Table 7.2.30-3. Summary of Calibration and QC Procedures for Method 314.0**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
314.0	Perchlorate	Multipoint calibration for all analytes (minimum 5 standards are recommended)	Initial calibration prior to sample analysis	option 1 linear- mean RSD ≤15%	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 2 linear - least squares regression $r > 0.995$		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification - Quality Control Sample (QCS)	Once per multipoint calibration, upon reestablishing calibration, quarterly	Instrument response within ± 10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
		Instrument Performance Check (IPC)	Daily, before sample analysis	Conductance within 10% of original value (original value within ± 10% of MCT)	Prepare fresh IPC solution	Apply R to all results for the sample
				$PD_{A/H} < 25\%$ , instrument response within ± 20% of expected response	Redetermine MCT or correct problem and reanalyze IPC	
				Retention time shifts < 5%, or overall retention time < 80% of original recorded value	Correct problem, clean or replace column	
		Initial calibration verification (ICCS)	Daily, before sample analysis or when eluent is changed	Instrument response within ± 25% of expected value using a standard at or below the RL	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration

**Table 7.2.30-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
314.0	Perchlorate	Calibration verification (CCCS/ECCS)	After every 10 samples (CCCS) and at the end of the analysis sequence (ECCS)	Instrument response within $\pm 15\%$ of expected response, alternately using separate mid and high level standards	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results in all samples since the last acceptable calibration verification
		Method blank (LRB)	One per analytical batch	Perchlorate must be = $\frac{1}{2}$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results in all samples in the associated analytical batch
		Pretreated laboratory reagent blank (LRB)	Required in any analytical batch which includes samples that have been pretreated to reduce the common anion levels	Perchlorate must be = $\frac{1}{2}$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results in all samples in the associated analytical batch
		Laboratory fortified blank (LFB)	One LFB per analytical batch following the ICCS	Instrument response within $\pm 15\%$ of expected response	Correct problem then reprep and analyze the LCS and all samples in the affected AFCEE batch	For all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Laboratory fortified matrix (LFM) and duplicate	One pair per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.30-2	none	For all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL

**Table 7.2.30-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
314.0	Perchlorate	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.30-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		MCT determination	At initial set-up, once per 12 month period	Calculate $PD_{A/H}$ for the perchlorate peak at increasing concentrations of mixed common anion solution. The MCT is the matrix conductance where the $PD_{A/H}$ exceeds 20%.	option 1 -least squares regression: plot $PD_{A/H}$ versus matrix conductance, ( $r^2 > 0.95$ ) option 2 - Use the conductance level of the highest mixed anion solution which yielded a $PD_{A/H}$ value < 20%	Samples cannot be analyzed without a valid MCT
		RL verification	At initial set-up, once per 12 month period	Instrument response within $\pm 30\%$ of expected response for a mixed common anion solution containing perchlorate at the RL and conductance within $\pm 10\%$ of the MCT	Lower the MCT by 10% and repeat the RL verification	Samples cannot be analyzed without a valid RL verification
		MDL study	At initial set-up, once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.30-1	none	Apply R to all results for in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### 7.2.31 Method RSK-175 –Soil Gases (Volatile Organics) in water

Soil gases in water are sampled and analyzed using method RSK-175. This method uses a high resolution GC coupled to one or more appropriate detectors (AFCEE requires the use of a mass-selective detector). The analytes detected and RLs for this method are listed in Table 7.2.31-1.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for BFB. The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50      15 percent to 40 percent of mass 95
- mass 75      30 percent to 60 percent of mass 95
- mass 95      base peak, 100 percent relative abundance
- mass 96      5 percent to 9 percent of mass 95
- mass 173     less than 2 percent of mass 174
- mass 174     greater than 50 percent of mass 95
- mass 175     5 percent to 9 percent of mass 174
- mass 176     greater than 95 percent, but less than 101 percent of mass 174
- mass 177     5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.31-2 and 7.2.31-3.

**Table 7.2.31-1. RLs for Method RSK-175**

Parameter/Method	Analyte	Water	
		RL	Unit
VOCs RSK-175	Methane	5	µg/L
	Ethane	5	µg/L
	Ethene	5	µg/L

**Table 7.2.31-2. QC Acceptance Criteria for Method RSK-175**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
RSK-175	Methane	60-120	≤ 20
	Ethane	65-115	≤ 20
	Ethene	65-115	≤ 20

**Table 7.2.31-3. Summary of Calibration and QC Procedures for Method RSK-175**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
RSK-175	Volatile Organics	Initial multipoint calibration minimum 3 standards	Initial calibration prior to sample analysis	%RSD for all calibration analytes $\leq$ 30% or linear regression correlation coefficient $r \geq 0.995$	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification (one point)	Daily, before sample analysis and every 12 hours of analysis time	All calibration analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.31-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify	Apply R to all results for all samples associated with the tune



**Table 7.2.31-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
RSK-175	Volatile Organics	ISs	Immediately after or during data acquisition for the calibration verification standard.	Retention time $\pm 30$ seconds from retention time of the mid-point std. in the ICAL.  EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for analytes associated with the IS
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.31-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE analytical batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.31-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

### 7.2.32 Method TO-14 -Volatile Organics in Ambient Air

Volatile organics in air are sampled and analyzed using method TO-14. This method uses a high resolution GC coupled to one or more appropriate detectors (AFCEE requires the use of a mass-selective detector). The analytes detected and RLs for this method are listed in Table 7.2.32-1.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for BFB. The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50 15 percent to 40 percent of mass 95
- mass 75 30 percent to 60 percent of mass 95
- mass 95 base peak, 100 percent relative abundance
- mass 96 5 percent to 9 percent of mass 95
- mass 173 less than 2 percent of mass 174
- mass 174 greater than 50 percent of mass 95
- mass 175 5 percent to 9 percent of mass 174
- mass 176 greater than 95 percent, but less than 101 percent of mass 174
- mass 177 5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.32-2 and 7.2.32-3.

**Table 7.2.32-1. RLs for Method TO-14**

Parameter/Method	Analyte	Air	
		RL	Unit
VOCs TO-14	1,1,1-TCA	0.8	ppbv
	1,2-DCA	0.6	ppbv
	1,2-Dibromoethane	0.6	ppbv
	Benzene	0.4	ppbv
	Carbon tetrachloride	2.1	ppbv
	Chloroform	0.3	ppbv
	m-Xylene	0.5	ppbv
	o-Xylene	1.1	ppbv
	p-Xylene	1.3	ppbv
	Styrene	0.4	ppbv
	TCE	1.0	ppbv

**Table 7.2.32-2. QC Acceptance Criteria for Method TO-14**

Method	Analyte	Accuracy Air (% R)	Precision Air (% RPD)
TO-14	1,1,1-TCA	72–125	≤ 20
	1,2-DCA	75–125	≤ 20
	1,2-Dibromoethane	74–125	≤ 20
	Benzene	75–127	≤ 20
	Carbon tetrachloride	72–125	≤ 20
	Chloroform	75–125	≤ 20
	m-Xylene	75–125	≤ 20
	o-Xylene	75–137	≤ 20
	p-Xylene	75–125	≤ 20
	Styrene	75–135	≤ 20
	TCE	75–125	≤ 20

**Table 7.2.32-3. Summary of Calibration and QC Procedures for Method TO-14**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
TO-14	Volatile Organics	Initial multipoint calibration (minimum 3 standards and humid zero air)	Initial calibration prior to sample analysis	%RSD for all calibration analytes $\leq 30\%$	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per three-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification (one point)	Daily, before sample analysis and every 12 hours of analysis time	All calibration analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.32-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify	Apply R to all results for all samples associated with the tune

**Table 7.2.32-3. Continued**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
TO-14	Volatile Organics	ISs	Immediately after or during data acquisition for the calibration verification standard.	Retention time $\pm 30$ seconds from retention time of the mid-point std. in the ICAL.  EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for analytes associated with the IS
		Method blank	One per analytical batch	No analytes detected $\geq$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.32-2	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE analytical batch	For specific analyte(s) in all samples in the associated analytical batch;  if the LCS %R > UCL, apply J to all positive results  if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

**Table 7.2.32-3. Concluded**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
TO-14	Volatile Organics	MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.32-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

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## **8.0 DATA REDUCTION, REVIEW, VERIFICATION, REPORTING, VALIDATION, AND RECORDKEEPING**

The data reduction, review, reporting, and validation procedures described in this section will ensure; (1) complete documentation is maintained, (2) transcription and data reduction errors are minimized, (3) the data are reviewed and documented, and (4) the reported results are qualified if necessary. Laboratory data reduction and verification procedures are required to ensure the overall objectives of analysis and reporting meet method and project specifications.

### **8.1 DATA REVIEW, VALIDATION, AND REPORTING REQUIREMENTS FOR SCREENING DATA**

The analysts shall perform a 100 percent review of the screening data. The screening data methods are identified in Table 6-1 of Section 6. All screening data shall be qualified with an *S* flag and shall be further qualified if critical calibration and QC requirements are not acceptable. The calibration, QC requirements, corrective action requirements, and flagging criteria required are shown in Table 6.2-1 in Section 6. Definitions of these data qualifiers are shown in Table 8.2-1. The flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. “S” designator flags shall be maintained in the final data qualification. When the data are reviewed and qualified, the analyst shall apply a final qualifier to any data that has been affected by multiple qualifiers. This final qualifier shall reflect the most severe qualifier that was applied to the data. The allowable final data qualifiers for screening data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are *SR*, *SJ*, *SB*, and *SU*. Therefore, the allowable final data qualifiers for screening data are *SR*, *SJ*, *SB*, *SU*, and *S*.

Screening data report packages shall be prepared for all field analyses as described in Section 8.8. The screening data shall be reported on the AFCEE screening data report forms (AFCEE Forms S-1 through S-3), as illustrated in Section 8.8. The prime contractor’s project manager shall review the entire screening data report package with the field records. The prime contractor (1) shall determine if the data quality objectives have been met, and (2) shall calculate the data completeness for the project. These results shall be included in the data package deliverable.



## **8.2 DATA REVIEW, VALIDATION, AND REPORTING REQUIREMENTS FOR DEFINITIVE DATA**

MDLs and sample results shall be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise. Soil samples shall have results reported on a dry weight basis. A wet weight aliquot of sample equivalent to the method specified dry weight aliquot of sample should be taken for analysis. Alternately, the lab may choose to use a consistent wet weight aliquot that is expected to be large enough to compensate for the moisture in the sample (e.g. 50% more) and use this as a consistent weight. RLs are NOT adjusted for sample moisture. If possible, samples should be analyzed undiluted and non-detects reported to the AFCEE specified RLs. RLs for minority constituents in highly contaminated samples may be adjusted for dilutions.

In each laboratory analytical section, the analyst performing the tests shall review 100 percent of the definitive data. After the analyst's review has been completed, 100 percent of the data shall be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria.

The definitive data methods are identified in Section 7.2. The calibration, QC requirements, corrective action requirements, and flagging criteria required for definitive data are shown in the tables in Section 7.2, and in summary Tables 8.2-2, 8.2-3, and 8.2-4. The flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Data qualifiers shall be added or, if applied by a software package, reviewed by the laboratory supervisor of the respective analytical section, after the first and second level of laboratory data reviews have been performed. Analytical batch comments shall be added to the first page of the definitive data report packages to explain any nonconformance or other issues. When data are qualified, the laboratory supervisor shall apply a final qualifier to any data that have been affected by multiple qualifiers. This final qualifier shall reflect the most severe qualifier that was applied to the data, i.e., all data will have only one data qualifying flag associate with it. The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of precedence, are *R*, *M*, *J*, *F*, *B*, and *U*. The definitions of the data qualifiers are shown in Table 8.2-1.

The one exception to these data flagging criteria rules applies to the tentatively identified compounds (TICs) that are identified only in the GC/MS methods. These TICs numerical results will always be qualified with one and only one flag for any reason, and that is the "T" flag.

The laboratory QA section shall perform a 100 percent review of 10 percent of the completed data packages, and the laboratory project manager shall perform a sanity check review on all the completed data packages.

The prime contractor's project manager shall review the entire definitive data report package, and with the field records, apply the final data qualifiers for the definitive data. The laboratory shall apply data qualifying flags to each environmental field QC sample, i.e., ambient blanks, equipment blanks, trip blanks, field duplicates, matrix spike (MS) samples, and matrix spike duplicate (MSD) samples. The prime contractor shall review the field QC samples and field logs, and shall then appropriately flag any of the associated samples identified with the field QC sample, as explained in Table 8.2-2 and 8.2-3. Each matrix spike sample shall only be qualified by the laboratory, while the prime contractor shall apply the final qualifying flag for a matrix effect to all samples collected from the same site as the parent sample or all samples showing the same lithologic characteristics as the MS/MSD.

The prime contractor (1) shall determine if the data quality objectives have been met, and (2) shall calculate the data completeness for the project. These results shall be included in the data package deliverable as described in Section 8.8. Contractual requirements for payment for laboratory services are beyond the scope of this document and may be different than the data validation requirements.

**Table 8.2-1 Data Qualifiers**

<b>Qualifier</b>	<b>Description</b>
J	The analyte was positively identified, the quantitation is an estimation.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
F	The analyte was positively identified but the associated numerical value is below the RL.
R	The data are rejected due to deficiencies in the ability to analyze the sample and meet QC criteria.
B	The analyte was found in an associated blank, as well as in the sample.
M	A matrix effect was present.
S	To be applied to all field screening data.
T	Tentatively identified compound (using GC/MS)

**Table 8.2-2. General Flagging Conventions**

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	R	All analytes in the sample
LCS	% R > UCL %R < LCL	J for the positive results J for the positive results, R for the nondetects	The specific analyte(s) in all samples in the associated AAB
Method Blank	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples in the associated AAB with results above the RL
Equipment Blank	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples with the same sampling date as the equipment blank
Field duplicates	Field duplicates > RLs AND RPD outside CL	J for the positive results R for the nondetects	The specific analyte(s) in all samples collected on the same sampling date
MS/MSD	MS or MSD % R > UCL OR MS or MSD % R < LCL OR MS/MSD RPD > CL	M for all results	The specific analyte(s) in all samples collected from the same site as the parent sample
Sample Preservation/ Collection	Preservation/collection requirements not met	R for all results	All analytes in the sample
Sample Storage	< 2°C or > 6°C or as required	J for the positive results R for the nondetects	All analytes in the sample

UCL = upper control limit      LCL = lower control limit      CL = control limit

	Criteria	Flag*
Quantitation	$\leq$ MDL	U
	> MDL < RL	F
	$\geq$ RL	as needed
	$\geq$ high std / linear range	J

\* Example 1: if the MDL is 0.04, the RL is 0.9 and the result is 0.03, the concentration reported on the result form would be 0.04 (the MDL) and the qualifier flag would be U.

Example 2: if the MDL is 0.04, the RL is 0.9 and the result is 0.07, the concentration reported on the result form would be 0.07 and the qualifier flag would be F.

Example 3: if the MDL is 0.04, the RL is 0.9 and the result is 1.2, the concentration reported on the result form would be 1.2 and the qualifier would be any flag needed because of a data quality problem (e.g., R, J, B, etc.).

**Table 8.2-3. Flagging Conventions Specific to Organic Methods**

QC Requirement	Criteria	Flag	Flag Applied To
Ambient Blank (VOC samples only)	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples with the same matrix and sampling date
Trip Blank (VOC samples only)	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples shipped in the same cooler as the blank
Initial Five Point Calibration (GC & HPLC methods)	Linearity criterion not met	R	The specific analyte(s) in all samples associated with the initial calibration
Initial Five Point Calibration (GC/MS methods)	SPCC or CCC criteria not met	R	All analytes in all samples associated with the initial calibration
	Linearity criterion not met	R	The specific analyte(s) in all samples associated with the initial calibration
Second Source Calibration Verification	CL exceeded	R	The specific analyte(s) in all samples associated with the second source calibration verification
Initial Daily Calibration Verification (GC & HPLC methods)	CL exceeded	R	The specific analyte(s) in all samples associated with the initial calibration verification
Calibration Verification (GC/MS methods)	SPCC or CCC criteria not met	R	All analytes in all samples associated with the calibration verification
	CL exceeded	R	The specific analyte(s) in all samples associated with the calibration verification
Calibration Verification (GC & HPLC methods)	CL exceeded	R	The specific analyte(s) in the sample associated with the continuing calibration verification
Retention time	Retention time of analyte outside of established retention time window	R	The specific analyte(s) in the sample
Surrogates	surrogate % R > UCL	J for the positive results	All analytes in the sample associated with the surrogate
	OR surrogate % R < LCL	J for the positive results R for the nondetects	
	OR surrogate recovery < 10%	R for all results	
Mass Spectrometer Tune	Ion abundance criteria not met	R for all results	All analytes in all samples associated with the tune

UCL = upper control limit      LCL = lower control limit      CL = control limit

**Table 8.2-3. Concluded**

QC Requirement	Criteria	Flag	Flag Applied To
Second Column/Second Detector Confirmation (GC & HPLC methods)	Not performed	R	All analytes $\geq$ RL
	Agreement between results not within $\pm 40\%$	J	All affected analytes
Internal Standard	Retention time not within $\pm 30$ seconds: EICP area not within -50% to +100% of last calibration verification	R	Apply R to all results for specific analytes associated with the IS
Lowest Calibration Standard	At or below RL in Initial Calibration	R	All results below the lowest calibration standard used
Tentatively Identified Compounds (TICs)		T	All TICs

**Table 8.2-4. Flagging Conventions Specific to Inorganic Methods**

QC Requirement	Criteria	Flag	Flag Applied To
Initial multipoint calibration	Correlation coefficient < 0.995	R	All results for specific analyte(s) for all samples associated with the initial calibration
Initial calibration verification/second source standard	CL exceeded	R	All results for specific analyte(s) for all samples associated with the calibration verification
Calibration blank	Analyte detected $\geq$ RL	B	All results for specific analyte(s) above the RL in all samples associated with the blank
Calibration verification (Instrument Check Standard)	CL exceeded	R	All results for specific analyte(s) in all samples since the last acceptable calibration verification
Interference check solution (ICS)	CL exceeded	R	All results for specific analyte(s) in all samples associated with the ICS
Dilution test	CL exceeded	J	Apply to all sample results if the new matrix check was not run or RPD $\geq$ 10%
Recovery test (GFAA methods)	CL exceeded	J	All samples in digestion batch if method of standard addition is not performed
Post digestion spike addition (ICP method)	CL exceeded	J	All sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
	% R < 10%	R	
Method of standard addition (GFAA methods)	Method of standard addition not done OR method of standard addition spike levels inappropriate OR correlation coefficient < 0.995	J	All positive sample results for specific analyte for all samples associated with the digestion batch

UCL = upper control limit      LCL = lower control limit      CL = control limit

### **8.3 QUALITY ASSURANCE REPORTS**

The laboratory QA staff shall issue QA reports to the laboratory management, laboratory supervisors and task leaders. These reports shall describe the results of QC measurements, performance audits, and systems audits, and confirmation sample comparisons performed for each sampling and analysis task. Quality problems associated with performance of methods, completeness of data, comparability of data including field and confirmatory data, and data storage shall be documented with the corrective actions that have been taken to correct the deficiencies identified.

### **8.4 ERPIMS ELECTRONIC DATA REPORTS**

The prime contractor shall provide an electronic deliverable report in the Environmental Restoration Program Information Management System (ERPIMS) format as specified by the SOW for the project.

ERPIMS is a data management system designed to accommodate all types of data collected for IRP projects. Specific codes and data forms have been developed to allow consistent and efficient input of information to the system. The database information shall be provided by the prime contractor via ASCII files in specified ERPIMS format on 3.5" floppy diskettes. The information transferred shall include all required technical data such as site information; well characteristics; and hydrogeologic, geologic, physical, and chemical analysis results. Electronic data reporting formats and requirements are given in the most current version of the *ERPIMS Data Loading Handbook*.

### **8.5 ARCHIVING**

Hardcopy and electronic data shall be archived in project files and on electronic archive tapes for the duration of the project or a minimum of five years, whichever is longer.

### **8.6 PROJECT DATA FLOW AND TRANSFER**

The data flow from the laboratory and field to the project staff and data users shall be sufficiently documented to ensure the data are properly tracked, reviewed, and validated for use.

### **8.7 RECORDKEEPING**

The laboratory shall maintain electronic and hardcopy records sufficient to recreate each analytical event conducted pursuant to the SOW. The minimum records the laboratory shall keep contain the following: (1) COC forms, (2) initial and continuing calibration records including standards preparation traceable to the original material and lot number, (3) instrument tuning records (as applicable), (3) method blank results, (4) IS results, (5) surrogate spiking records and results (as applicable), (6) spike and spike duplicate records and results, (7) laboratory records, (8) raw data,

including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports, (9) corrective action reports, (10) other method and project required QC samples and results, and (11) laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

## **8.8 HARDCOPY DATA REPORTS FOR SCREENING AND DEFINITIVE DATA**

The hardcopy data reports shall conform to the formats identified in this section.

A screening data report package shall consist of the following AFCEE forms: COC, S-1, S-2, and S-3.

A definitive data inorganic report package shall consist of the following AFCEE forms: COC, I-1, I-2, I-3, I-4, I-5, I-6, I-7, I-8 and I-9 for each AAB with inorganic analyses performed.

A definitive data organic report package shall consist of the following AFCEE forms: COC, O-1, O-2, O-3 or O-3A, O-4, O-5 or O-5A, O-6, O-7, O-8, O-9 and O-10 for each AAB with organic analyses performed.

A definitive data wet chemistry report package shall consist of the following AFCEE forms: COC, W-1, W-2, W-3, W-4, W-5, W-6, W-7, W-8, and W-9 for each AAB with wet chemistry analyses performed.

Exceptions to these report forms are as follows: for mercury analysis, form I-3A shall be substituted for form I-3 in the inorganic report package; for cyanide analysis, form I-3B shall be substituted for form I-3 in the inorganic report package; for GC/MS analyses, forms O-3A and O-5A shall be used and form O-11 shall be added to the organic report package.



## INSTRUCTIONS FOR COMPLETING AFCEE REPORT FORMS

The following instructions shall be used in completing the AFCEE report forms for screening and definitive data. The bold lettering identifies the fields on the AFCEE report form.

Use as many sheets as necessary. Sheets may be duplicated with only those sections necessary to be completed filled out (i.e., you do not have to duplicate previously reported information from one sheet to the next). Sequentially number the sheets at the bottom of the page if more than one sheet is necessary.

**\*Reporting Dilutions\*** Justification for diluting samples shall be provided in the comments section on the appropriate form (I-2, O-2 or W-2). If the result for any analyte is outside the calibration range (i.e., greater than the highest calibration standard), the sample shall be diluted appropriately and reanalyzed. Results from the undiluted and diluted sample shall be reported on the appropriate form (I-2, O-2 or W-2). The results of the analysis of the diluted sample shall be reported with the dilution noted on the report form and the MDL and RL adjusted for the dilution.

### ALL INORGANIC , ORGANIC AND WET CHEM FORMS

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Lab Name:** enter the laboratory name (e.g., Garland Labs, Inc.)

**Contract #:** enter the Air Force contract number and delivery order number under which the analytical work is being performed (e.g., F21625-94-D-8005/0001)

**Comments:** enter any comments

### FORM I-1

**Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/SPACECOM)

**Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

FORM I-1 (continued)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID

FORM I-2

This form is completed for all environmental samples including the MD and MSD.

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID

**Matrix:** enter the sample matrix (e.g., water, soil)

**% Solids:** enter the % solids

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the sample results

**Date Received/Prepared/Analyzed:** enter the appropriate dates in the format DD-MMM-YY (e.g., 3 Jun 96)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg dry weight)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**MDL:** enter the laboratory derived method detection limit

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Concentration:** enter the numeric result

**Dilution:** enter the dilution (if applicable) (e.g., 1:5)

**Qualifier:** enter the qualifier flag (see QAPP Sections 7 and 8)

FORM I-3

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 96)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

**RF1, RF2, RF3:** enter the response factor corresponding to the standard with the same number

**Std 1, Std2, Std3:** enter the concentration of the standard

**r:** enter the correlation coefficient

**Q:** enter a "\*" for all corresponding correlation coefficients that were not acceptable as per QAPP Section 7

FORM I-3A (Mercury analyses only)

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 96)

**Initial Calibration ID:** enter the unique identifying number given to this initial calibration event

FORM I-3A (continued)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**RF1, RF2, RF3, RF4, RF5:** enter the response factor corresponding to the standard with the same number

**Std 1, Std 2, Std 3, Std 4, Std 5:** enter the concentration of the standard

**r:** enter the correlation coefficient

**Q:** enter a “\*” for all corresponding correlation coefficients that were not acceptable as per QAPP Section 7

FORM I-3B (Cyanide analyses only)

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY  
(e.g., 3 Jun 96)

**Initial Calibration ID:** enter the unique identifying number given to this initial calibration event

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**RF1, RF2, RF3, RF4, RF5, RF6:** enter the response factor corresponding to the standard with the same number

**Std 1, Std 2, Std 3, Std 4, Std 5, Std 6:** enter the concentration of the standard

**r:** enter the correlation coefficient

**Q:** enter a “\*” for all corresponding correlation coefficients that were not acceptable as per QAPP Section 7

FORM I-3B (continued)

**Expected:** enter the expected result (i.e., the concentration of the calibration material).

**Found:** enter the measured result.

**%D:** enter the per cent difference between the expected and found

FORM I-4

**AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results

**2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., 2S960603)

**CCV #1 ID:** enter the unique identification number for the first CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-1)

**CCV #2 ID:** enter the unique identification number for the second CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-2)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**Expected:** enter the expected result (i.e., the concentration of the calibration material).

**Found, Found 1, Found 2:** enter the measured result. Found 1 corresponds to the first CCV run, Found 2 corresponds to the second CCV run, etc.

FORM I-4 (continued)

**%D:** enter the per cent difference between the expected and found

**Q:** enter a "\*" for any %D that was not acceptable as per QAPP Section 7

FORM I-5

**AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Initial Calibration Blank ID:** enter the identification number for the calibration blank (the same ID number will be found in the run sequence log, e.g., CB960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the initial calibration blank results

**Method Blank ID:** enter the unique identifying number given to the method blank (the same ID number will be found in the run sequence log, e.g., MB960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results

**CCB #1 ID:** (used for 6010B analysis) enter the identification number for the first CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-1)

**CCB #2 ID:** (used for 6010B analysis) enter the identification number for the second CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-2)

**CCB #3 ID:** (used for 6010B analysis) enter the identification number for the third CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-3)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**Initial Calibration Blank:** enter a numeric result for the calibration blank

**Continuing Calibration Blank 1:** enter a numeric result for the first continuing calibration blank run

FORM I-5 (continued)

**Continuing Calibration Blank 2:** enter a numeric result for the second continuing calibration blank run

**Continuing Calibration Blank 3:** enter a numeric result for the third continuing calibration blank run

**Method Blank:** enter a numeric result for the method blank

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Q:** enter a "\*" for any calibration or method blank analytes that were not acceptable as per QAPP Section 7

FORM I-6

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log e.g., LCS960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in LCS material)

**Found:** enter the measured result of the LSC analytes

**%R:** enter the per cent recovery

**Control Limits:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter a "\*" for any %R that was not acceptable as per QAPP Section 7

FORM I-7

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**% Solids:** enter the % solids of the parent field sample

**Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)

**MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log e.g., MS960603)

**MSD ID:** enter the unique identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log e.g., MSD960603)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**Parent Sample Result:** enter the numeric result of the parent sample. If an analyte was not detected above the MDL, leave this column blank

**Spike Added:** enter the amount of spike added to the parent sample

**Spiked Sample Result:** enter the numeric result of the MS

**%R:** enter the per cent recovery

**Duplicate Spiked Sample Result:** enter the numeric result of the MSD

**%RPD:** enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

**Control Limits %R:** enter the control limits required to be met (see QAPP Section 7)

**Control Limits %RPD:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter the qualifier flag as needed (see QAPP Sections 7 and 8)



FORM I-8

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Date Collected:** enter the date the sample was taken in the field in the format DD-MMM-YY (e.g., 6 Jun 96)

**Date Received:** enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

**Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

**Max. Holding Time:** enter the maximum allowable holding time in days (see QAPP Section 5)

**Time Held:** enter the time in days elapsed between the date collected and the date analyzed

**Q:** enter a "\*" for any holding times that were greater than the maximum allowable holding time as per QAPP Section 5

FORM I-9

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed

**Date Analysis Started:** enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 6 Jun 96)

**Time Analysis Started:** enter the time the sample analysis was started in 24-hour format (e.g., 0900, 2130)

**Date Analysis Completed:** enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 6 Jun 96)

FORM I-9 (continued)

**Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour format  
(e.g., 0900, 2130)

FORM O-1

**Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/  
SPACECOM)

**Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS,  
MSD, field duplicate and field blanks)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that  
corresponds to the Field Sample ID

FORM O-2

This form is completed for all environmental samples including the MD and MSD.

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP  
for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS,  
MSD, field duplicate and field blanks)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that  
corresponds to the Field Sample ID

**Matrix:** enter the sample matrix (e.g., water, soil)

**% Solids:** enter the % solids

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event  
used in the determination of the sample results

**Date Received/Prepared/Analyzed:** enter the appropriate dates in the format DD-MMM-YY  
(e.g., 3 Jun 96)

FORM O-2 (continued)

**Concentration Units:** enter the appropriate units (i.e., µg/L or mg/kg dry weight)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**MDL:** enter the laboratory derived method detection limit

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Concentration:** enter the numeric result

**Dilution:** enter the dilution (if applicable) (e.g., 1:5)

**Confirm:** enter the numeric result from the confirmation column/detector

**Qualifier:** enter the qualifier flag as needed (see QAPP Section 7)

**Surrogate:** enter the name of the surrogate(s) used

**Recovery:** enter the per cent recovery of the surrogate

**Control Limits:** enter the control limits for the recovery of the surrogate (see QAPP section 7)

**Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used

FORM O-3 and 3A

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 96)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event

FORM O-3 and 3A (continued)

**Concentration Units:** enter the appropriate units (i.e., µg/L or mg/kg)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.  
(On form 3A, some analyte names already appear on the form as provided, leave those analytes in that order.)

**RF1, RF2, RF3, RF4, RF5, RF6, RF7:** enter the response factor corresponding to the standard with the same number (RF6 and RF7 are used for non-linear calibrations)

**Std 1, Std 2, Std 3, Std 4, Std 5, Std 6, Std 7:** enter the concentration of the standard (Std 6 and Std 7 are used for non-linear calibrations)

**%RSD:** enter the per cent relative standard deviation of the response factors

**Mean %RSD:** enter the mean of the RSDs of all analytes for those analytes not using a least squares regression or non-linear calibration

**r:** (optional) if least squares regression is used for the calibration of an analyte, enter the correlation coefficient

**COD:** (optional) if a non-linear calibration is used for the calibration of an analyte, enter the coefficient of determination

**Q:** enter a "\*" for any calibration that was not acceptable as per QAPP Section 7 and for any RFs not meeting minimum requirements for SPCCs and/or CCCs.

FORM O-4

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration event pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the second source calibration verification results

FORM O-4 (continued)

**2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., 2S960603)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.

**Expected:** enter the expected result (i.e., the concentration of the calibration material).

**Found:** enter the measured result.

**%D:** enter the per cent difference between the expected (i.e., the concentration of the second source calibration material) and measured result

**Q:** enter a “\*” for any % D that was not acceptable as per QAPP Section 7

FORM O-5 and O-5A

**AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results

**ICV ID:** enter the unique identification number for the ICV such that the ICV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., ICV960603-1)

**CCV #1 ID:** enter the unique identification number for the CCV run after the first 12 hours of operation such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-1)

**CCV #2 ID:** enter the unique identification number for the CCV run after the second 12 hours of operation such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-2)

FORM O-5 and O-5A (continued)

**Analyte:** enter all analyte names in the same order as listed in the tables in QAPP Section 7.  
(On form O-5A, some analyte names already appear on the form as provided, leave those analytes in that order.)

**RF:** (form O-5A) enter the response factor for the SPCCs only

**% D:** enter the per cent difference

**% D or % drift:** (form O-5) enter the per cent difference if using RFs or % drift if using CFs

**Q:** enter a "\*" for any % drift that was not acceptable as per requirements in QAPP Section 7

FORM O-6

**AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Concentration Units:** enter the appropriate units (i.e., µg/L or mg/kg)

**Method Blank ID:** enter the unique identification number for the method blank (the same ID number will be found in the run sequence log, e.g., MB960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results

**Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the QAPP)

**Method Blank:** enter a numeric result for the method blank

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Q:** enter a "\*" for any method blank analyte result that was not acceptable as per QAPP Section 7

**Surrogate:** enter the name of the surrogate(s) used

**Recovery:** enter the per cent recovery of the surrogate

FORM O-7

**Control Limits:** enter the control limits for the recovery of the surrogate (see QAPP section 7)

**Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., LCS960603)

**Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$ )

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results

**Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the QAPP)

**Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in the LCS)

**Found:** enter the measured result of the LSC analytes

**%R:** enter the per cent recovery

**Control Limits:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter a "\*" for any % R that was not acceptable as per QAPP Section 7

**Surrogate:** enter the name of the surrogate(s) used

**Recovery:** enter the per cent recovery of the surrogate

**Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used

FORM O-8

**Concentration Units:** enter the appropriate units (i.e., µg/L or mg/kg)

**Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)

**% Solids:** enter the % solids

**MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log, e.g., MS960603)

**MSD ID:** enter the identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log, e.g., MSD960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the MS/MSD results

**Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the QAPP)

**Parent Sample Result:** enter the result of the parent sample. If an analyte was not detected above the MDL, leave this column blank.

**Spike Added:** enter the amount of spike added to the parent sample

**Spiked Sample Result:** enter the numeric result of the MS

**%R:** enter the per cent recovery

**Duplicate Spiked Sample Result:** enter the numeric result of the MSD

**%RPD:** enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

**Control Limits %R:** enter the control limits required to be met (see QAPP Section 7)

**Control Limits %RPD:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter the qualifier flag as needed (see QAPP Sections 7)



FORM O-9

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Date Collected:** enter the date the sample was taken in the field in the format DD-MMM-YY (e.g., 3 Jun 96)

**Date Received:** enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)

**Date Extracted:** enter the date the sample was extracted by the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)

**Max. Holding Time E:** enter the maximum allowable holding time in days until the sample is extracted (if applicable - see QAPP Section 5)

**Time Held Ext.:** enter the time in days elapsed between the date collected and the date extracted (if applicable)

**Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)

**Max. Holding Time A:** enter the maximum allowable holding time in days until the sample is analyzed (see QAPP Section 5)

**Time Held Anal.:** enter the time in days elapsed between the date collected and the date analyzed

**Q:** enter a "\*" for any holding time (Max. Holding Time E, or Max. Holding Time A, or Time Held Anal.) that was greater than the maximum holding time that was not acceptable as per QAPP Section 5

FORM O-10

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

FORM O-10 (continued)

**Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed

**Date Analysis Started:** enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 3 Jun 96)

**Time Analysis Started:** enter the time the sample analysis was started in 24-hour format (e.g., 0900, 2130)

**Date Analysis Completed:** enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 3 Jun 96)

**Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 2130)

FORM O-11

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Compound:** enter BFB or DFTPP as appropriate

**Injection Date/Time:** enter the date (in the format DD-MMM-YY) and time (in 24-hour format) of the performance check

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the MS/MSD results

**Mass:** enter the mass of the ion used for tuning (see QAPP Section 7)

**Ion Abundance Criteria:** enter the criteria for the specific mass (see QAPP Section 7)

**% Relative Abundance:** enter the per cent relative abundance as the result of the tune

**Q:** enter a "\*" for any % relative abundance results that was not acceptable as per QAPP Section

FORM W-1

**Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/  
SPACECOM)

**Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS,  
MSD, field duplicate and field blanks)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that  
corresponds to the Field Sample ID

FORM W-2

This form is completed for all environmental samples including the MD and MSD.

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP  
for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS,  
MSD, field duplicate and field blanks)

**Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that  
corresponds to the Field Sample ID

**Matrix:** enter the sample matrix (e.g., water, soil)

**% Solids:** enter the % solids

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event  
used in the determination of the sample results

**Date Received/Prepared/Analyzed:** enter the appropriate dates in the format DD-MMM-YY  
(e.g., 3 Jun 96)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg dry weight)

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of  
the QAPP)

FORM W-2 (continued)

**MDL:** enter the laboratory derived method detection limit

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Concentration:** enter the numeric result

**Dilution:** enter the dilution (if applicable) (e.g., 1:5)

**Qualifier:** enter the qualifier flag (see QAPP Sections 7 and 8)

FORM W-3

**AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 96)

**Initial Calibration ID:** enter the unique identifying number given to this initial calibration event

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

**RF1, RF2, RF3:** enter the response factor corresponding to the standard with the same number

**Std 1, Std2, Std3:** enter the concentration of the standard

**r:** enter the correlation coefficient

**Q:** enter a “\*” for any correlation coefficients that were not acceptable as per QAPP Section 7

FORM W-4

**AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results

**2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., 2S960603)

**ICV ID:** enter the unique identification number for the ICV such that the ICV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., ICV960603)

**CCV #1 ID:** enter the unique identification number for the first CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-1)

**CCV #2 ID:** enter the unique identification number for the second CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-2)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

**Expected:** enter the expected result (i.e., the concentration of the calibration material)

**Found, Found 1, Found 2:** enter the measured result. Found 1 corresponds to the first CCV run, Found 2 corresponds to the second CCV run, etc.

**%D:** enter the per cent difference between the expected and found

**Q:** enter a "\*" for any %D that was not acceptable as per QAPP Section 7

FORM W-5

**AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Calibration Blank ID:** enter the identification number for the calibration blank (the same ID number will be found in the run sequence log, e.g., CB960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration blank results

**Method Blank ID:** enter the identification number for the method blank (the same ID number will be found in the run sequence log, e.g., MB960603)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

**Calibration Blank:** enter a numeric result for the calibration blank

**Method Blank:** enter a numeric result for the method blank

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Q:** enter a "\*" for any calibration or method blank analyte that was not acceptable as per QAPP Section 7

FORM W-6

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log e.g., LCS960603)

FORM W-6 (continued)

**Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

**Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in LCS material)

**Found:** enter the measured result of the LCS analyte

**%R:** enter the per cent recovery

**Control Limits:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter a “\*” for any %R that was not acceptable as per QAPP Section 7

FORM W-7

**% Solids:** enter the % solids

**Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)

**MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log e.g., MS960603)

**MSD ID:** enter the unique identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log e.g., MSD960603)

**Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)

**Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

FORM W-7 (continued)

**Parent Sample Result:** enter the numeric result of the parent sample. If an analyte was not detected above the MDL, leave this column blank

**Spike Added:** enter the amount of spike added to the parent sample

**Spiked Sample Result:** enter the numeric result of the MS

**%R:** enter the per cent recovery

**Duplicate Spiked Sample Result:** enter the numeric result of the MSD

**%RPD:** enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

**Control Limits %R:** enter the control limits required to be met (see QAPP Section 7)

**Control Limits %RPD:** enter the control limits required to be met (see QAPP Section 7)

**Q:** enter the qualifier flag as needed (see QAPP Sections 7 and 8)

FORM W-8

**AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the AFCEE QAPP for a definition of a batch)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Date Collected:** enter the date the sample was taken in the field in the format DD-MMM-YY (e.g., 6 Jun 96)

**Date Received:** enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

**Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

**Max. Holding Time:** enter the maximum allowable holding time in days (see QAPP Section 5)

**Time Held:** enter the time in days elapsed between the date collected and the date analyzed



FORM W-8 (continued)

**Q:** enter a "\*" for any holding time that was greater than the maximum allowable holding time as per QAPP Section 5

FORM W-9

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed

**Date Analysis Started:** enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 6 Jun 96)

**Time Analysis Started:** enter the time the sample analysis was started in 24-hour format (e.g., 0900, 2130)

**Date Analysis Completed:** enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 6 Jun 96)

**Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 2130)

FORM S-1

**Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/SPACECOM)

**Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Signature:** signature of person completing data package

**Name:** name of person completing data package

**Date:** enter the date the in the format DD-MMM-YY (e.g., 6 Jun 96)

FORM S-1 (continued)

**Title:** title of person completing data package

FORM S-2

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

**Matrix:** enter the sample matrix (e.g., water, soil)

**Date Analyzed:** enter the appropriate dates in the format DD-MMM-YY (e.g., 3 Jun 96)

**Units:** enter the appropriate units (e.g., µg/L, mg/kg, degrees C)

**Analyte/Test:** enter the name of the analyte or test performed (e.g., pH)

**MDL:** enter the method detection limit if applicable

**RL:** enter the project AFCEE reporting limit as stated in the QAPP or approved variance for each analyte

**Result:** enter the result

**Qualifier:** enter the qualifier needed (see QAPP Sections 7 and 8)

FORM S-3

**Units:** enter the appropriate units (e.g., µg/L, mg/kg, degrees C)

**Analyte/Test:** enter the name of the analyte or test performed (e.g., pH)

**Sample Result:** enter the result of the sample

**Duplicate Sample Result:** enter the result of the duplicate sample

**%D or %RPD:** enter the per cent or difference relative per cent difference between the sample and duplicate as appropriate

**Acceptance Criteria:** enter the acceptance criteria required to be met (see QAPP Section 6)

**Q:** enter a “\*” for any % D or % RPD that was not acceptable as per QAPP Section 6

MDL FORM

**Matrix:** enter the sample matrix (e.g., water, soil)

**Analysis Date:** enter the date (or inclusive dates if performed over a period of days) the MDL was performed in the format DD-MMM-YY (e.g., 6 Jun 96)

**Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)

**Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the QAPP)

**Amt. Spiked:** enter the amount of spike added to the matrix

**Replicate 1,2,3,4,5,6,7:** enter the result of the replicate

**Std. Dev.:** enter the standard deviation of the seven replicates

**MDL:** enter the calculated MDL

CHAIN OF CUSTODY FORM

**COC#:** enter a unique number for each chain of custody form

**Ship to:** enter the laboratory name and address

**Carrier:** enter the name of the transporter (e.g., FedEx) or handcarried

**Airbill#:** enter the airbill number or transporter tracking number (if applicable)

**Project Name:** enter the project name (e.g., Banks AFB RI/FS)

**Sampler Name:** enter the name of the person collecting the samples

**Sampler Signature:** signature of the person collecting the samples

**Send Results to:** enter the name and address of the prime contractor

**Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

CHAIN OF CUSTODY FORM (continued)

**Date:** enter the year and date the sample was collected in the format M/D (e.g., 6/3)

**Time:** enter the time the sample was collected in 24-hour format (e.g., 0900)

**Matrix:** enter the sample matrix (e.g., water, soil)

**Pres:** enter the preservative used (e.g., HNO<sub>3</sub>) or “none”

**Filtered/Unfilt.:** enter “F” if the sample was filtered or “U” if the sample was not filtered

**# of Containers:** enter the number of containers (i.e., jars, bottles) associated with the sample

**MS/MSD:** enter “X” if the sample is designated the MD/MSD

**Analyses Requested:** enter the method name of the analysis requested (e.g., SW6010B)

**Comments:** enter comments

**Sample Condition Upon Receipt at Laboratory:** enter any problems with the condition of any sample(s)

**Cooler Temperature:** enter the internal temperature of the cooler, upon opening, in degrees C

**Special Instructions/Comments:** enter any special instructions or comments

**Released by: (SIG):** enter the signature of the person releasing custody of the samples

**Company Name:** enter the company name employing the person releasing/receiving custody

**Received by: (SIG):** enter the signature of the person receiving custody of the samples

**Date:** enter the date in the format M/D/YY (e.g., 6/3/96) when the samples were released/  
received

**Time:** enter the time in 24-hour format (e.g., 0900) when the samples were released/received

Prime Contractor: \_\_\_\_\_

[illegible]

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Title: \_\_\_\_\_

Concentration Units (mg/L or mg/kg dry weight): \_\_\_\_\_

[illegible]

AFCEE FORM I-2 Page \_\_\_\_ of \_\_\_\_

Instrument ID: \_\_\_\_\_ Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

AFCEE FORM I-3

AFCEE  
 INORGANIC ANALYSES DATA SHEET 3A  
 MERCURY INITIAL MULTIPOINT CALIBRATION

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
 Initial Calibration ID: \_\_\_\_\_ Concentration Units (mg/L or mg/kg): \_\_\_\_\_

Analyte	Std 1	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	r	Q
Mercury												

r = correlation coefficient

Comments:

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AFCEE  
 INORGANIC ANALYSES DATA SHEET 3B  
 CYANIDE INITIAL MULTIPOINT CALIBRATION

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
 Initial Calibration ID: \_\_\_\_\_ Concentration Units (mg/L or mg/kg): \_\_\_\_\_

Analyte	Std 1	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	Std 6	RF 6	r	Q
Cyanide														

r = correlation coefficient

	Expected	Found	%D	Q
High Distilled Standard				
Low Distilled Standard				

Comments:

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Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

AFCEE FORM I-4 Page \_\_\_\_ of \_\_\_\_

Method Blank ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

[illegible]

AFCEE FORM I-5 Page \_\_\_\_ of \_\_\_\_

Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

AFCEE FORM I-6

Parent Field Sample ID: \_\_\_\_\_ MS ID: \_\_\_\_\_ MSD ID: \_\_\_\_\_

[illegible]

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Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

[illegible]

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Prime Contractor: \_\_\_\_\_

[illegible]

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Title: \_\_\_\_\_



AFCEE  
ORGANIC ANALYSES DATA SHEET 2  
RESULTS

Analytical Method: \_\_\_\_\_ Preparatory Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Field Sample ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_ Matrix: \_\_\_\_\_

% Solids: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

Date Received: \_\_\_\_\_ Date Prepared: \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

Concentration Units (ug/L or mg/kg dry weight): \_\_\_\_\_

Analyte	MDL	RL	Concentration	Dilution	Confirm	Qualifier

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments:

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Concentration Units (ug/L or mg/kg): \_\_\_\_\_

[illegible]

AFCEE FORM O-3A Page \_\_\_\_ of \_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_

AFCEE FORM O-3A Page \_\_\_\_ of \_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_

[illegible]

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[illegible]

AFCEE FORM O-3 Page \_\_\_\_ of \_\_\_\_

2nd Source ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

[illegible]

AFCEE FORM O-4 Page \_\_\_\_ of \_\_\_\_

ICV ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

[illegible]

AFCEE FORM O-5A Page \_\_\_\_ of \_\_\_\_

ICV ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

[illegible]

AFCEE FORM O-5 Page \_\_\_\_ of \_\_\_\_



AFCEE  
ORGANIC ANALYSES DATA SHEET 6  
BLANK

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_ Method Blank ID: \_\_\_\_\_

Initial Calibration ID: \_\_\_\_\_

Analyte	Method Blank	RL	Q

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments:

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Concentration Units (ug/L or mg/kg): \_\_\_\_\_

[illegible]

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments:

Parent Field Sample ID: \_\_\_\_\_ MS ID: \_\_\_\_\_ MSD ID: \_\_\_\_\_

[illegible]

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Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

[illegible]

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AFCEE  
ORGANIC ANALYSES DATA SHEET 11  
INSTRUMENT PERFORMANCE CHECK  
(BFB or DFTPP)

Analytical Method: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Compound: \_\_\_\_\_ Injection Date/Time: \_\_\_\_\_

Initial Calibration ID: \_\_\_\_\_

Mass	Ion Abundance Criteria	% Relative Abundance	Q

No mention of DDT breakdown or of benzidine and pentachlorophenol tailing. Should these be included?

Prime Contractor: \_\_\_\_\_

Lab Sample ID

[illegible][illegible]

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Title: \_\_\_\_\_

Concentration Units (mg/L or mg/kg dry weight): \_\_\_\_\_

[illegible]

AFCEE FORM W-2 Page \_\_\_\_ of \_\_\_\_



Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

Comments:

2nd Source ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

[illegible]

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Method Blank ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

[illegible]

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Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

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Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

[illegible]

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AFCEE  
SCREENING DATA PACKAGE

Analytical Method: \_\_\_\_\_

Contract #: \_\_\_\_\_

Base/Command: \_\_\_\_\_

Prime Contractor: \_\_\_\_\_

Field Sample ID

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Signature: \_\_\_\_\_

Name: \_\_\_\_\_

Date: \_\_\_\_\_

Title: \_\_\_\_\_



Concentration Units (ug/L, mg/kg dry weight or °C): \_\_\_\_\_

[illegible]

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Units: \_\_\_\_\_

[illegible]

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Concentration Units (mg/L or mg/kg): \_\_\_\_\_

[illegible]

## AFCEE CHAIN OF CUSTODY RECORD

COC#: \_\_\_\_\_

Ship to:		Project Name:		Send Results to:	
		Sampler Name:			
Carrier:		Airbill #:			

Field Sample ID	Date 19__	Time	Matrix	Pres	Filtered/ Unfilt.	# of Containers	MS/ MSD	Analyses Requested										Comments

Sample Condition Upon Receipt at Laboratory:	Cooler temperature:
Special Instructions/Comments:	

#1 Released by: (Sig)	Date:	#2 Released by: (Sig)	Date:	#3 Released by: (Sig)	Date:
Company Name:	Time	Company Name:	Time	Company Name:	Time
#1 Received by: (Sig)	Date	#2 Received by: (Sig)	Date	#3 Received by: (Sig)	Date
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:

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## **9.0 SYSTEMS AND PERFORMANCE AUDITS, PERFORMANCE EVALUATION PROGRAMS, MAGNETIC TAPE AUDITS, AND TRAINING**

Technical systems and performance audits shall be performed as independent assessments of sample collection and analysis procedures. Audit results will be used to evaluate the ability of an analytical contractor to (1) produce data that fulfill the objectives established for the program, (2) comply with the QC criteria, and (3) identify any areas requiring corrective action. The systems audit is a qualitative review of the overall sampling or measurement system, while the performance audit is a quantitative assessment of a measurement system. Audit guidance can be found in the *HQ AFCEE Technical Services Quality Assurance Program*, current version. Full data validation is also a quantitative check of the analytical process, where all documentation and calculations are evaluated and verified. Data validation is discussed in Section 8.

### **9.1 PROJECT AUDITS**

#### **9.1.1 State/Federal Project Audits**

Audits by various state and federal agencies are commonly conducted for the laboratories that will analyze project samples. Audit reports from these agencies shall be reviewed by the prime contractor to determine whether data produced by the analytical contractor shall fulfill the objectives of the program.

Audit findings shall be transmitted from the laboratory to the prime contractor and to AFCEE. The prime contractor shall review the audit findings and provide a written report to AFCEE. This report shall include the recommended corrective actions or procedures to correct the deficiencies identified during the state/federal audits(s). The audit results and discussion shall be incorporated into the QA report for each sampling effort.

#### **9.1.2 Technical Systems Audits**

A technical systems audit is an on-site, qualitative review of the sampling or analytical system to ensure that the activity is being performed in compliance with the Sampling and Analysis Plan (SAP) specifications. Sampling and field procedures, and the analytical laboratories shall be audited by the prime contractor at the beginning of the project. In addition, a laboratory systems audit may be performed by AFCEE if previous audit reports indicate corrective actions are outstanding, a recent audit has not been conducted, or quality concerns have arisen based upon the use of that laboratory for other projects. The laboratory systems audit results will be used to assess the prime contractor's oversight and to review laboratory operation and ensure the technical procedures and documentation are in place and operating to provide data that fulfill the project objectives and to ensure outstanding corrective actions have been addressed.

Critical items for a laboratory or field systems audit include: (1) sample custody procedures, (2) calibration procedures and documentation, (3) completeness of data forms, notebooks, and

other reporting requirements, (4) data review and validation procedures, (5) data storage, filing, and record keeping procedures, (6) QC procedures, tolerances, and documentation, (7) operating conditions of facilities and equipment, (8) documentation of training and maintenance activities, (9) systems and operations overview, and (10) security of laboratory automated systems.

Critical items for a sampling systems audit include: (1) calibration procedures and documentation for field equipment, (2) documentation in field logbooks and sampling data sheets, (3) organization and minimization of potential contamination sources while in the field, (4) proper sample collection, storage, and transportation procedures, and (5) compliance with established COC and transfer procedures.

After each on-site audit, a debriefing session will be held for all participants to discuss the preliminary audit results. The auditor will then complete the audit evaluation and submit an audit report including observations of the deficiencies and the necessary recommendations for corrective actions to the prime contractor. Compliance with the specifications presented in the SAP will be noted and noncompliance or deviations shall be addressed in writing by the prime contractor to AFCEE with corrective actions and a time frame for implementation of the corrective actions. Follow-up audits will be performed prior to completion of the project to ensure corrective actions have been taken.

### **9.1.3 Project-Specific Performance Evaluation Audits**

Performance audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting project-specific performance evaluation (PE) samples for analysis for each analytical method used in the project. The prime contractor shall submit project specific PE samples once per quarter per project. The project-specific PE samples are selected to reflect the expected range of concentrations for the sampling program. The performance audit answers questions about whether the measurement system is operating within control limits and whether the data produced meet the analytical QA specifications.

The project-specific PE samples are made to look as similar to field samples as possible and are submitted as part of a field sample shipment so that the laboratory is unable to distinguish between them and project samples. This approach ensures unbiased sample analysis and reporting by the laboratory.

The critical elements for review of PE results include: (1) correct identification and quantitation of the PE sample analytes, (2) accurate and complete reporting of the results, and (3) measurement system operation within established control limits for precision and accuracy.

The concentrations reported for the PE samples shall be compared to the known or expected concentrations spiked in the samples. The percent recovery shall be calculated and the results assessed according to the accuracy criteria for the LCS presented in Section 7. If the accuracy criteria are not met, the cause of the discrepancy shall be investigated and a second PE sample

shall be submitted. The prime contractor shall notify the project staff, AFCEE, and agencies of the situation at the earliest possible time and the prime contractor shall keep AFCEE up to date regarding corrective actions and subsequent PE sample results.

#### **9.1.4 Magnetic Tape Audits**

Magnetic tape audits involve the examination of the electronic media used by the analytical laboratory and by the prime contractor to collect, analyze, report, and store data. These audits are used to assess the authenticity of the data generated, and assess the implementation of good automated laboratory practices. AFCEE may perform magnetic tape audits of the laboratories or of the prime contractors when warranted by project PE results, on-site audit results, or by other state/federal investigations.

#### **9.1.5 Performance Evaluation Sample Programs**

All laboratories shall participate in the U.S. EPA PE Water Supply and Water Pollution Studies programs or equivalent programs for state certifications. Satisfactory performance in these nonproject-specific PE programs also demonstrate proficiency in methods used to analyze AFCEE samples. The laboratory shall document the corrective actions to unacceptable PE results to demonstrate resolution of the problems.

### **9.2 TRAINING**

Training shall be provided to all project personnel to ensure compliance with the health and safety plan and technical competence in performing the work effort. Documentation of this training shall be maintained in the records of the contracted organizations.



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## **10.0 PREVENTIVE MAINTENANCE**

A preventive maintenance program shall be in place to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to unexpected component failure. In implementing this program, efforts are focused in three primary areas: (1) establishment of maintenance responsibilities, (2) establishment of maintenance schedules for major and/or critical instrumentation and apparatus, and (3) establishment of an adequate inventory of critical spare parts and equipment.

### **10.1 MAINTENANCE RESPONSIBILITIES**

Maintenance responsibilities for equipment and instruments are assumed by the respective facility managers. The managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to the prescribed protocols.

### **10.2 MAINTENANCE SCHEDULES**

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted as needed. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/mass spectrometry instruments, AA spectrometers, and analytical balances).

### **10.3 SPARE PARTS**

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. The inventory includes those parts (and supplies) that are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, the contractor shall maintain an in-house source of backup equipment and instrumentation.

### **10.4 MAINTENANCE RECORDS**

Maintenance and repair of major field and laboratory equipment shall be recorded in field or laboratory logbooks. These records shall document the serial numbers of the equipment, the

person performing the maintenance or repairs, the date of the repair, the procedures used during the repair, and proof of successful repair prior to the use of the equipment.

## **11.0 CORRECTIVE ACTION**

Corrective actions, if necessary, shall be completed once. If acceptance criteria were not met and a corrective action was not successful or corrective action was not performed, apply the appropriate flagging criteria. Requirements and procedures for documenting the need for corrective actions are described in this section.

### **11.1 CORRECTIVE ACTION REPORT**

Problems requiring corrective action in the laboratory shall be documented by the use of a corrective action report. The QA coordinator or any other laboratory member can initiate the corrective action request in the event QC results exceed acceptability limits, or upon identification of some other laboratory problem. Corrective actions can include reanalysis of the sample or samples affected, resampling and analysis, or a change in procedures, depending upon the severity of the problem.

### **11.2 CORRECTIVE ACTION SYSTEM**

A system for issuing, tracking, and documenting completion of formal Recommendations for Corrective Action (RCA) exists for addressing significant and systematic problems. Recommendations for corrective actions are issued only by a member of the QA group, or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of laboratory or project operations. An RCA requires a written response from the party to whom the RCA was issued. A summary of unresolved RCAs is included in the monthly QA report to management. The report lists all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. An RCA requires verification by the QA group that the corrective action has been implemented before the RCA is considered to be resolved. In the event there is no response to an RCA within 30 days, or if the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved.

#### **11.2.1 Manual Integration**

Manual integration is not to be a routine procedure for the purpose of meeting QA/QC acceptance criteria. It is to be done only rarely as a corrective action measure. An example would be when there are co-eluting compounds and those compounds can not be separated by the instrument. Manual integration would be appropriate to identify the peaks. When manual integration is used the following procedures are to be implemented for documenting the event and to conduct training for consistency in performing the manual integration.

- AFCEE requires the laboratory or section SOP to include instructions for the analyst to document any required manual integrations associated with the initial/continuing calibration. The SOP is to require the following hard copy documentation:
  - A “before” and “after” hard copy for the manual integration, with the reason, date and signature of the analyst;
  - Review and approval for the manual integration by the Section supervisor and the QAO.
- AFCEE requires that technical guidelines be developed for manual integration. Topics covered must include under what conditions manual integrations are to be initiated and what constitutes technically acceptable manual integration events. This will ensure consistency when manual integrations are performed.

## **12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT**

At a minimum, the QA coordinator of the laboratory shall prepare a summary report quarterly of the status of the project, of QA/QC problems, corrective actions taken, and unresolved RCAs with recommended solutions for management. The report shall also include results from all PE samples, audit findings, and periodic data quality assessments. This report shall be available for review by AFCEE auditors upon request.